ENVIRONMENTAL PROTECTION AGENCY
OFFICE OF ENFORCEMENT

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Compliance Evaluation
And
Wastewater Characterization
South Charleston Sewage Treatment Company
South Charleston, West Virginia

NATIONAL ENFORCEMENT INVESTIGATIONS CENTER
DENVER, COLORADO
AND
REGION III PHILADELPHIA

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Environmental Protection Agency
Office of Enforcement
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COMPLIANCE EVALUATION
AND
WASTEWATER CHARACTERIZATION

SOUTH CHARLESTON SEWAGE TREATMENT COMPANY
SOUTH CHARLESTON, WEST VIRGINIA

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National Enforcement Investigations Center - Denver
and
Region III - Philadelphia

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I. INTRODUCTION

The South Charleston Sewage Treatment Company (SCSTC),* South Charleston, West Virginia, is a joint venture between Union Carbide and the City of South Charleston. The Company treats approximately 19,000 m^3/day (5 mgd) of Union Carbide South Charleston process wastewaters and 8,000 m^3/day (2 mgd) of municipal wastewaters. The industrial and municipal wastewaters are treated separately [Figure 1]. The effluents from domestic and industrial treatment units combine and discharge to the Kanawha River through Outfall 001.

The Kanawha Valley contains numerous industrial plants engaged in the production of organic and/or inorganic chemicals. The passage of the Toxic Substance Control and Resources Conservation and Recovery Acts in 1976 focused attention on the need to control the discharges of toxic substances. Large volumes of such wastes are produced and disposed of in the Kanawha Valley, from which toxic substances could then be released to the environment.

On January 10, 1978, the Environmental Protection Agency (EPA) Region III requested that the National Enforcement Investigations Center (NEIC) investigate the SCSTC to identify and quantify toxic chemicals discharged to the Kanawha River and to determine compliance with the National Pollutant Discharge Elimination System (NPDES)** permit limitations. NEIC conducted a detailed plant inspection and subsequent field survey.

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* The treatment facility is referred to by Company personnel as the South Charleston Waste Treatment Works.

** NPDES: National Pollutant Discharge Elimination System, Public Law 92-500, Sec. 402 of the Federal Water Pollution Control Act as amended in 1972, and subsequently Sec. 402 of the Clean Water Act as amended in 1977.

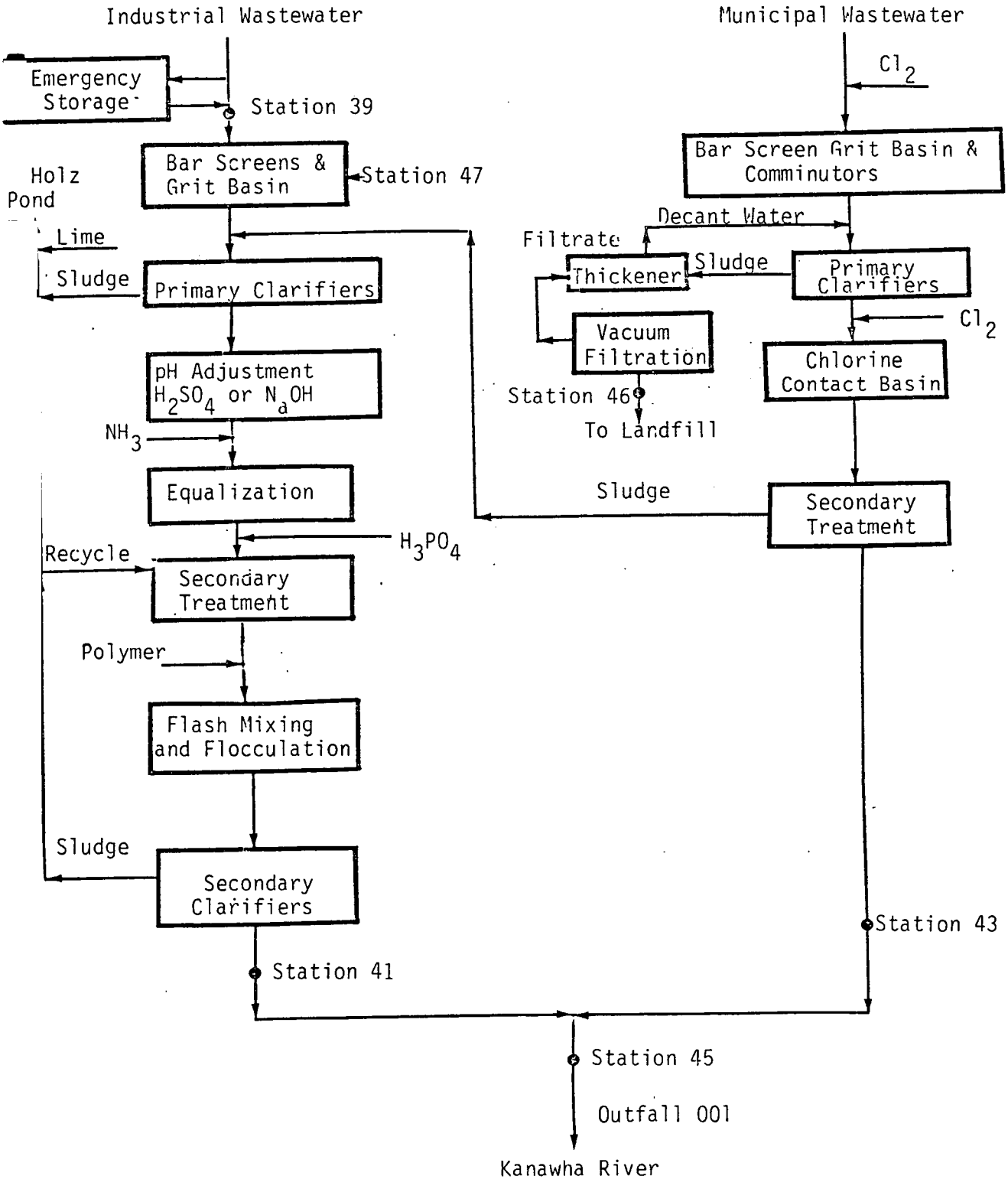

Figure 1. Schematic of South Charleston Sewage Treatment Company

The objectives of the April 1978 plant inspection were to:

1. inspect treatment units and evaluate operation practices
2. evaluate NPDES self-monitoring procedures.

The objectives of the August 1978 survey were to:

1. measure wastewater flows
2. determine compliance with NPDES permit effluent limitations
3. collect wastewater samples for organic characterization.

Organic compounds identified during the wastewater characterization were evaluated to determine potential health effects.

II. SUMMARY AND CONCLUSIONS

SUMMARY OF INVESTIGATIONS

NEIC personnel inspected the SCSTC facility in April 1978. Plant operations were discussed in detail with Company personnel. Treatment units were inspected and operational practices evaluated. Self-monitoring procedures including sample collection, flow monitoring, sample analysis, bioassay procedures, and discharge monitoring reports (DMRs) were also evaluated.

A monitoring survey was conducted at this facility August 15 to 21, 1978. Seven 24-hour flow-weighted composite samples were collected to determine compliance with NPDES permit effluent limitations. Composite samples from the industrial influent, industrial effluent, domestic effluent and final discharge were analyzed for organic compounds. Each organic compound was searched in the Registry of Toxic Effects and Chemical Substances and the Toxline data bases to obtain toxic information. Bioassay and mutagenicity tests were also conducted on the treatment plant effluent.

CONCLUSIONS

Fecal coliform samples are not collected from the final discharge. These organisms are being monitored in the chlorine contact basin and domestic effluents. Company data show that while fecal coliform organisms in the chlorine contact basin effluent are low (0 to 503/100 ml log mean) the domestic effluent is high (80 to 7,000/100 ml log mean).

Data from the flow meter installed on Outfall 001 in August 1978 and subsequent DMR data based on these results are not reliable. Instantaneous measurements using the lithium chloride dilution technique showed that at the time of the survey the meter was recording flow 26 to 38% lower than actual. Pollutant loads calculated based on these flows would also be low.

In general, chemical analyses are performed by SCSTC personnel according to EPA-approved methods. Fecal coliform analyses were being performed by the membrane filter procedures. Unless comparability of an alternate method can be demonstrated, the 5-tube MPN procedure is recommended for self-monitoring. Vinyl chloride monomer testing has produced low recoveries on spiked samples. Additional work is required on test procedures to ensure adequate recovery of spiked samples.

In general bioassay procedures were adequate. Discrepancies observed include: a) not starting test within 8 hours as recommended by Standard Methods®, b) using city dechlorinated tap water was used as dilution water instead of Kanawha River water, c) not running tests in duplicate, and d) aerating samples throughout the 96-hour test period. It is advisable, through not required, that the laboratory use a constant temperature water bath to maintain test temperature rather than depending on ambient air temperature.

A review of the DMRs showed that the Company violated one or more of its permit limitation for the period October 1977 through September 1978. BOD, TSS, COD, NH_3, chloride and pH limitations were exceeded during this period.

Survey data show that the SCSTC is capable of meeting permit limitations except for fecal coliform. The 7-day average concentrations were 20 mg/l BOD, 330 mg/l COD, 24 mg/l TSS, 15 mg/l TKN, 8.4 mg/l NH_3, 260 mg/l chloride and 12 µg/l phenol. These values are

only 24, 49, 30, 29, 56, 65 and 24% of their respective permit limitations. Fecal coliform densities ranged from 790 to 130,000/100 ml with a geometric mean of 12,000/100 ml. The permit requires that the 7-day fecal coliform organism geometric mean not exceed 400/100 ml.

Analyses of the WWTF effluent discharge show that the facility is discharging priority pollutants.* Maximum concentrations of the 6 pollutants identified were 80 µg/l zinc, 200 µg/l nickel, 15 µg/l chloroform, 41 µg/l methylene chloride, 4 µg/l tetrachloroethene and 6 µg/l 1,1,1-trichloroethane.

Primary domestic sludge and industrial grit containing priority pollutants are being buried in non-secure landfills (city of South Charleston and Filmont, respectively). Both the sludge and grit contained As, Cr, Cu, Ni, Pb, Zn and Hg with concentrations ranging from 2.2 to 2,500 µg/l. The industrial grit also contained isophorone at 120 µg/g.

The SCSTC effluent is not acutely toxic to fish. Bioassay results show 95% survival for 96-hours in 100% effluent.

Mutagenic and potential carcinogenic substances are being discharged from Outfall 001. Each of the 3 samples collected from this discharge demonstrated a mutagenic activity ratio greater than 2.5 which correlates closely (>90% probability) with inducement of cancer in laboratory animals.

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* For explanation of priority pollutants see Section V.

III. TREATMENT PLANT DESCRIPTION

SCSTC is operated and maintained by the City of South Charleston and is staffed with a superintendent, assistant superintendent, clerk, four shift supervisors, eight plant operators, 2 vacuum filter operators, and two maintenance personnel. The laboratory is staffed with one chemist who oversees the analytical tests conducted by plant operators.*

The municipal wastewater secondary treatment system, designed for a population equivalent of 37,500, receives wastes from the city of South Charleston (population of approximately 23,000). The wastewater first receives preliminary treatment consisting of prechlorination, bar screening, grit removal and comminutors. Materials removed by the bar screen and grit chamber are hauled to the city of South Charleston (SC) landfill.

The wastewater then enters two 1,050 m^3 (277,300 gal) primary clarifiers operated in parallel. The primary sludge is pumped to a thickener, concentrated, treated with ferric chloride and lime, vacuum filtered and buried (approximately 3,200 kg - 7,000 lb/day) at the SC landfill. Thickener decant water is returned to the clarifiers and water removed in the vacuum filters is discharged back to the thickener. Effluent from the primary clarifiers is chlorinated to maintain approximately 0.5 ppm residual chlorine. The chlorinated wastewater then enters two Aero Accelators (activated sludge process) operated in parallels.** These units, 4,280 m^3 (1.13 mg) capacity each, provide

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* Each operator is responsible for conducting analyses necessary to run the treatment units, as well as those analyses required by the NPDES permit.

** During both the inspection and survey, only one unit was being operated.

both aeration and secondary clarification. Each unit has a 150 HP agitator and a compressed air diffuser which maintains a D.O. concentration of 6 to 7 ppm. Waste-activated sludge is pumped into the industrial primary clarifier influent. The overflow from the Aero Accelators combines with the industrial effluent (discussed below) and is discharged into the Kanawha River through Outfall 001.

Industrial wastewater from UCSC, including supernatant from Holz Pond and plant domestic wastes, are routed to the SCSTC through an open redwood flume. Concentrated process wastes and spills are diverted from the flume into a 3,800 m^3 emergency storage tank. These wastes are slowly returned to the treatment plant via the redwood flume [Figure 1].

The industrial wastewaters pass through two grit basins (each 3 x 6 m - 10 x 19 ft) into two primary clarifiers (each 1,050 m^3 - 277,300 gal) operated in parallel. The industrial grit is buried at the Filmont landfill which is owned and operated by Union Carbide. As previously noted, the municipal waste-activated sludge combines with the influent to these primary clarifiers. Sludge removed from the primary clarifiers is treated with lime and pumped to Holz Pond. Supernatant from this pond is returned to the treatment facility via the redwood flume.

The primary clarified wastewater is neutralized with either NaOH or H_2SO_4 and ammonia is added* as a nitrogen source. The wastewater is then discharged into the three 3,800 m^3 (1 mg) equalization tanks operated in parallel.

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* One kg of NH_3 is added for every 40 kg of BOD removed in the aeration basins.

Wastewater from the equalization tanks is discharged at a controlled rate into a large aeration basin which has a capacity of 24,400 m^3 (6.45 mg). H_3PO_4 is added* to the wastewater as it enters the basin. The basin is equipped with seventeen - 100 HP surface aerators and five - 65 HP bottom mixers. Plant officials try to maintain D.O. levels of 2 to 3 ppm at the surface and 0.5 ppm at the bottom of the basin.

Polymer is added to the aeration basin effluent which then flows to flash mixing and flocculation. The wastewater then enters three secondary clarifiers, operated in parallel. Each clarifier has a capacity of 2,420 m^3 (640,500 gal). The clarifier effluent combine with the municipal treated effluent and discharge to the Kanawha River through Outfall 001. Secondary sludge is combined with primary sludge and lime and pumped to Holz Pond.

Municipal wastewater influent flow is measured by a 0.46 m (1.5 ft) Parshall flume. The flow is continuously recorded and totalized. The flume is located downstream of a small lift station which contributes less than 10% of the domestic flow. As the lift station pumps do not operate continuously, the flow chart has numerous peaks.

Industrial influent flows are measured with a 0.61 m (2 ft) Parshall flume and continuously recorded and totalized. Visual observations showed that the wastewater is diverging** as it enters the converging section causing turbulance through the flume. As a result, the flow recording trace is almost 2.5 cm (1 in) wide. The midpoint of the trace is used to determine the flow.

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* The wastewater is deficient in phosphorus. Plant personnel add one kg of H_3PO_4 for every 220 kg of BOD removed in the basin.

** The wastewater enters the flow device through a conduit which has a diameter smaller than the upstream converging section.

Wastewater entering the industrial aeration basin is measured with a 0.76 m (2.5 ft) Palmer-Bowles flume. Magnetic flow-meters are used to measure industrial primary clarifiers underflow and waste-activated sludge.

During the April inspection, flow rates for the combined discharge (Outfall 001) were being calculated based on the sum of the industrial and municipal influents, less the amount of industrial clarifier underflow and waste-activated sludge being pumped to Holz Pond. Subsequent to the initial inspection, a Model 250 March McBirney flow meter was installed to measure and record the volume of wastewater discharged through Outfall 001.

IV. SURVEY METHODS

Self-monitoring practices, including flow measurement and sampling techniques, analytical and bioassay procedures and DMR results, were evaluated April 11 to 12, 1978 at the SCSTC. Sampling was performed from August 15 to 22, 1978 to determine compliance with NPDES permit WV 0023117 [Table 1] and characterize wastewater. Samples were collected from the industrial influent, industrial effluent, municipal effluent, total plant discharge (Outfall 001) to the Kanawha River, primary domestic sludge and industrial grit [Figure 1]. Chain-of-Custody procedures were followed for the collection of the samples* and for laboratory analyses. Flow verification procedures and sampling techniques are discussed in Appendix B.

The Parshall Flumes installed on the domestic and industrial influents were checked prior to sampling and found to be installed according to recommendations of the Water Measurement Manual. The flow recording devices and totalizers were compared to flume head measurements and found to be operating properly. The flow measurement devices on the influent to the aeration basin and total discharge (Outfall 001) were checked using the lithium chloride tracer dilution technique [Appendix B].

Sample aliquots were manually collected hourly and continually composited on a flow-weighted basis for all parameters except volatile

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* The samples sent to Denver August 20 were received without a lock on the ice chest. The samples were packaged in either plastic containers or plastic sacks. There did not appear to be any tampering with the samples prior to arrival at the NEIC laboratory.

Table 1

NPDES FINAL PERMIT LIMITATIONS
SOUTH CHARLESTON SEWAGE TREATMENT COMPANY
SOUTH CHARLESTON, WEST VIRGINIA

Parameter[a]	Average Effluent Concentrations 30 Consecutive Day Period (mg/l)	7 Consecutive Day Period (mg/l)	30 Consecutive[b] Day Period lbs/day	kg/day
Biochemical Oxygen Demand (5-day) May-Oct	53	80	5,300	2,400
Nov-Apr	84	126	8,400	3,810
Suspended Solids	53	79	5,300	2,400
Fecal Coliforms	200[c]	400[c]		
pH	within limits of 6.0-9.0 at all times			
Chemical Oxygen Demand				
May-Oct	450	680	45,000	20,400
Nov-Apr	580	870	58,000	26,400
Phenols	0.03	0.05	3.0	1.4
Kjeldahl Nitrogen				
May-Oct	35	52	3,500	1,550
Nov-Apr	40	60	4,000	1,820
Ammonia Nitrogen				
May-Oct	10	15	1,000	450
Nov-Apr	15	20	1,500	680
Chlorides	200	400	20,000	9,100
Temperature	Maximum of 43.3°C (110°F)			

a In addition, the Company is required quarterly to determine vinyl chloride monomer and toxicity. Toxicity is to be monitored by bioassays.

b The 30 consecutive day average quantity of effluent discharged from the wastewater treatment facility shall not exceed 15.8 million gallons per day (mgd) or 59,800 cubic meters per day.

c per 100 ml.

organics, direct aqueous injection and fecal coliform which were collected three times each day. All samples were collected over the period 12 midnight to 12 midnight which corresponds to permit requirements. The parameters monitored and the sample type for each station are shown in Table 2. All samples were analyzed by the procedures in Appendices C and D.

Flow-through bioassays were conducted August 15 to 19 on the plant effluent (Outfall 001). The wastewater was continuously pumped directly from the outfall to the bioassay laboratory on an equal-volume basis. Dilution water was obtained from the Kanawha River at a point approximately 3.2 km (2 miles) upstream of the mouth of the Elk River. A discussion of the bioassay procedures is contained in Appendix E.

Analyses for mutagenic activity were performed on three 24-hour flow-proportional composite samples from SCSTC effluent (Station 45). The Ames Bacterial Assay for Mutagenicity was performed on liquid sample concentrates using the agar plate incorporation method, as described by Ames, et al.[1] The Standard Ames Test determined mutagenic activity through use of bacteria as indicator organisms; this information correlates closely ($\geq$90% probability) with inducement of cancer in laboratory animals by organic compounds.[2,3,4]

Acidic and basic sample extracts were prescreened for mutagenic activity using four standard Salmonella tester strains, TA 98, TA 100, TA 1535 and TA 1537. Samples were first tested individually and then subjected to metabolic activation by addition of rat-liver homogenate [Appendix F].

Table 2

DESCRIPTION OF SAMPLING STATIONS
SOUTH CHARLESTON SEWAGE TREATMENT COMPANY (SCSTC)

Station[a]	Description	Type of Sample	Parameter[b]
39	SCSTC Industrial Influent	24-hour Composite	Chloride; COD; NH_3; TKN; phenol; organics
		Grab	Volatile organics; direct aqueous injection
41	SCSTC Industrial Effluent	24-hour Composite	BOD; TSS; chloride; COD; NH_3; TKN; phenol; organics
		Grab	Volatile organics; fecal coliform[c]
43	SCSTC Domestic Effluent	24-hour Composite	BOD; TSS; chloride; COD; NH_3; TKN; phenol; organics
		Grab	Volatile organics; fecal coliform
45	SCSTC Final Effluent (Outfall 001)	24-hour Composite	BOD; TSS; chloride; COD; NH_3; TKN; metals; organics; mutagens[d]
		Grab	Volatile organics; direct aqueous injection; fecal coliform bacteria
46	SCSTC Primary Sludge[e]	Grab	Trace metals and organics
47	SCSTC Industrial Grit	Grab	Trace metals and organics

a Figure 1 shows station location.
b Temperature and pH were measured periodically at all stations.
c Grab samples collected 3 times each day for this parameter.
d Mutagen samples were collected three times during the survey.
e Primary sludge and industrial grit samples were collected once during the survey.

V. SURVEY RESULTS

PERFORMANCE AUDIT

Evaluations of self-monitoring procedures practiced by SCSTC were conducted. Plant personnel were interviewed and equipment and procedures observed. The NEIC evaluation indicated the following procedures deviated from prescribed/recommended techniques.

Flow Monitoring

SCSTC did not measure the wastewater discharged through Outfall 001 until August 1978. Flow data reported on DMRs prior to August were determined based on domestic influent and industrial influent, both measured with Parshall flumes. These flumes are installed properly and, even with the turbulence previously noted, provide reliable data. As noted, the feed to the industrial aeration basin from the equalization tanks is controlled by the operators. The amount treated is based on the flow received the previous day. Thus, the daily flow adjustment lags by at least one day. On a 30-day average, the values should be reliable.

The Company installed a Marsh McBirney Model 250 meter in August to measure and continuously record the flow as required by the NPDES permit.

Bioassay Procedures

The Company bioassay facilities are maintained at the Union Carbide Technical Center in South Charleston. The facility is environmentally

controlled and properly equipped for bioassay testing. The bioassays and the associated chemical tests are performed according to Standard Methods except as noted below:

1. The bioassay tests do not always commence within eight hours after sample collection as recommended by Standard Methods.

2. Dechlorinated city tap water is used as dilution water rather than Kanawha River water as required by the NPDES permit.

3. The bioassay tests are not done in duplicate as recommended by Standard Methods.

4. All bioassays are aerated throughout the 96-hour test period. Aeration should be discontinued except in cases where BOD or COD are sufficiently high that adequate dissolved oxygen concentrations cannot be maintained.

5. The laboratory depends on controlled ambient air temperature to maintain a constant test temperature. It is advisable, though not required, that a constant temperature water bath be used to maintain constant test temperature.

Analytical Procedures

The membrane filter procedure for monitoring bacteria is being used. The membrane filter technique usually yields low and variable recovery from chlorinated wastewaters.

Initial testing for the vinyl chloride monomer in the effluent has produced low recoveries on the spiked samples. Emphasis should be placed on improving percentage of recoveries on samples spiked with vinyl chloride monomer.

Sampling

The company has an in-plant wastewater monitoring program including total carbon analyzers. Composite samplers are not refrigerated, however, the data are used to aid in-plant operations. Effluent samples (Outfall 001) are manually collected, composited and refrigerated. Plant personnel stated that a new refrigerated automatic flow-proportional sampler is to be installed on Outfall 001.

Fecal coliform organism samples are collected from the chlorine contact basin effluent and the Aero Accelator effluent. The NPDES permit limits fecal coliform organisms in the total plant effluent (Outfall 001), not at these intermediate points.

NPDES EFFLUENT LIMITATION COMPLIANCE

Results of verifying the Marsh McBirney flow meter (Outfall 001) with lithium chloride data are tabulated below.

Date	Time	NEIC Lithium Chloride Flow m^3/day	NEIC Lithium Chloride Flow mgd	SCSTC Meter Flow m^3/day	SCSTC Meter Flow mgd
18	1725	32,400	8.6	28,800	6.3
19	2043	31,000	8.2	20,100	5.3
20	2041	34,100	9.0	21,900	5.8
21	0841	41,200	10.9	28,800	7.6
Avg.	--	34,700	9.2	24,900	6.3

These data show that the Marsh McBirney meter was recording value 26 to 38% lower than the actual flow. The sum of the domestic influent plus industrial aeration basin influent flows, however, were comparable to Outfall 001 lithium chloride results. Therefore, the daily flow data reported in Table 3 were calculated based on this summation.

Table 3

SUMMARY FIELD MEASUREMENTS AND ANALYTICAL DATA
SOUTH CHARLESTON SEWAGE TREATMENT COMPANY

Parameters (August) →	15	16	17	18	19	20	21	Average
Station 45 DISCHARGE TO KANAWHA RIVER (OUTFALL 001)								
Flow								
m^3/day x 10^3	35.4	36.0	29.0	35.0	28.5	32.6	33.0	32.8
mgd	9.36	9.51	7.65	9.24	7.54	8.63	8.73	8.67
Temperature °C Range	25-27	26-29	26-28	25-28	27-29	27-28	25-26	
pH Range	6.1-7.9	7.5-7.8	6.4-7.9	7.5-7.9	7.5-7.8	7.4-8.1	7.7-8.0	
BOD								
mg/l	18	12	12	34	22	24	21	20
kg/day	640	430	350	1,200	630	780	690	670
lb/day	1,400	950	770	2,600	1,400	1,700	1,500	1,500
COD								
mg/l	160	240	370	360	420	390	360	330
kg/day	5,700	8,600	11,000	13,000	12,000	13,000	12,000	11,000
lb/day	12,000	19,000	24,000	28,000	26,000	28,000	26,000	23,000
TSS								
mg/l	19	18	23	57	21	18	15	24
kg/day	670	650	670	2,000	600	590	500	810
lb/day	1,500	1,400	1,500	4,400	1,300	1,300	1,100	1,800
TKN								
mg/l	18	18	15	15	13	12	11	15
kg/day	640	650	430	520	370	390	360	480
lb/day	1,400	1,400	960	1,200	820	860	800	1,100
NH_3-N								
mg/l	9.1	13	8.9	10	5.6	6.8	5.2	8.4
kg/day	320	470	260	350	160	220	170	280
lb/day	710	1,000	570	770	350	490	380	610
Chloride								
mg/l	260	330	340	280	200	190	190	260
kg/day	9,200	12,000	9,800	9,800	5,700	6,200	6,300	8,400
lb/day	20,000	26,000	22,000	22,000	13,000	14,000	14,000	19,000
Phenol								
µg/l	12	9	16	10	16	8	13	12
kg/day	0.43	0.32	0.46	0.35	0.46	0.26	0.43	0.39
lb/day	0.94	0.71	1.0	0.77	1.0	0.58	0.95	0.85

Effluent data collected August 15 to 21 show that all results except those for fecal coliform bacteria [Table 3 and 4] are less than permit limits. The seven-day average concentration [Table 3] for BOD (20 mg/l), COD (330 mg/l), TSS (24 mg/l), TKN (15 mg/l), NH_3 (8.4 mg/l), chloride (260 mg/l) and phenol (12 µg/l) were only 25, 49, 30, 29, 56, 65 and 24% of their respective permit limitations. Fecal coliform bacteria densities in the effluent ranged from 790 to 130,000/100 ml with a geometric mean bacterial density of 12,000/100 ml [Table 4]. This high fecal coliform bacterial density exceeds the NPDES permit limitation of $\leq$400/100 ml for each seven day consecutive period.

DMR data for October 1977 through September 1978 [Table 5] show that the average BOD, TSS and TKN concentrations during the August 15 to 21, 1978 study were atypically low. For example, reported average BOD concentrations ranged from 35 to 326 mg/l, approximately 2 to 16 times greater than survey results (20 mg/l). These DMRs also show that the SCSTC exceeded permit limitations 5, 6, 2, 3, 9 and 1 months respectively for BOD, TSS, COD, NH_3, chloride and pH.

WASTEWATER CHARACTERIZATION

Industrial Influent (Station 39)

Evaluation of the Parshall flume by NEIC personnel showed that the influent flows were within ±10% of actual when the flow recorder trace was read at the midpoint. During the survey, 16,600 to 20,400 m^3/day or process wastewaters were received from UCSC.

The NEIC sampled and analyzed the UCSC process wastewater, industrial influent, for a variety of parameters [Table 2]. The wastewater contained an average of 2,700 mg/l (52,000 kg/day) COD, 36 mg/l (690 kg/day) TKN, 5.7 mg/l (110 kg/day) NH_3, and 300 mg/l (5,500 kg/day)

Table 4

SUMMARY OF FECAL COLIFORM BACTERIA DENSITIES
SOUTH CHARLESTON SEWAGE TREATMENT COMPANY
SOUTH CHARLESTON, WEST VIRGINIA

Station No.	Description	No. of Samples	Fecal Coliform Bacteria MPN/100 ml Maximum	Minimum	Geometric Mean
41	South Charleston, W. Va. Sewage Treatment Co. Industrial Effluent	21	4,900	20	84
43	South Charleston, W. Va. Sewage Treatment Co. Domestic Effluent	21	350,000	3,300	27,000
45	South Charleston, W. Va. Sewage Treatment Co. Final Effluent (Outfall 001)	22	130,000	790	12,000

Table 5

SUMMARY OF DISCHARGES MONITORING REPORTS[a]
SOUTH CHARLESTON SEWAGE TREATMENT COMPANY
October 1977 - September 1978

Parameter	Oct	Nov	Dec	Jan	Feb	Mar	Apr	May	Jun	Jul	Aug	Sep
Flow												
m^3/day	21.0	21.3	25.2	32.7	27.5	29.8	24.3	32.3	27.6	26.1	30.0	29.5
mgd	5.56	5.63	6.67	8.65	7.27	7.86	6.42	8.53	7.30	6.89	7.92	7.8
BOD												
mg/l	74	81	217	177	326	76	89	42	50	35	40	46
kg/day	1,591	1,727	5,591	5,794	8,970	2,261	2,162	1,356	1,381	913	1,199	1,358
lb/day	3,508	3,807	12,326	12,777	19,778	4,985	4,768	2,990	3,046	2,012	2,644	2,994
TSS												
mg/l	76	76	138	147	254	53	65	28	37	45	48	46
kg/day	1,591	1,591	3,727	4,812	6,989	1,577	1,579	904	1,022	1,173	1,439	1,358
lb/day	3,508	3,508	8,217	10,611	15,410	3,476	3,482	1,993	2,254	2,587	3,172	2,994
COD												
mg/l	439	430	785	579	990	513	496	332	314	220	286	256
kg/day	9,273	9,091	20,273	18,955	27,239	15,260	12,051	10,718	8,675	5,737	8,573	7,557
lb/day	20,443	20,042	44,694	41,795	60,061	33,649	26,573	23,633	19,128	12,649	18,902	16,663
Phenol												
mg/l	0.016	0.011	0.019	0.02	0.021	0.02	0.011	0.013	0.015	0.013	0.013	0.016
kg/day	0.30	0.23	0.48	0.65	0.58	0.59	0.27	0.42	0.41	0.34	0.39	0.47
lb/day	0.66	0.51	1.06	1.44	1.27	1.31	0.59	0.92	0.91	0.75	0.86	1.04
TKN												
mg/l	26	21	33	28	37	24	23	27	24	20	20	26
kg/day	564	427	850	917	1,018	714	559	872	663	519	602	768
lb/day	1,243	941	1,874	2,021	2,245	1,574	1,232	1,922	1,462	1,144	1,328	1,692
NH_3-N												
mg/l	12	8	3	2	6	8	14	17	16	6.2	6.7	10.6
kg/day	255	159	77	65	165	238	340	549	442	162	201	313
lb/day	562	350	170	144	364	525	750	1,210	975	356	443	690
Chlorides												
mg/l	199	669	597	287	377	433	169	314	182	221	232	539
kg/day	4,895	13,218	15,363	9,395	10,373	12,880	4,106	10,137	5,028	5,752	6,954	15,911
lb/day	10,792	29,140	33,870	20,717	22,872	28,401	9,054	22,351	11,087	12,684	15,333	35,084
pH Range	7.0-7.6	6.9-7.7	7.0-9.4	7.0-7.6	6.6-8.0	6.8-7.8	7.2-7.9	7.4-8.6	7.4-8.1	7.1-8.2	6.0-9.0	7.5-8.2
Temperature												
max °C	22.2	15.6	11.7	10.6	7.8	13.9	18.9	22	25	28	32	27

a lb/day and m^3/day not reported by the plant; values computed by NEIC.

chloride and 380 µg/l (7 kg/day) phenolic compounds [Table 6]. The pH fluctuated from 2.3 to 12.0 during the survey.

Characterization of the process wastewater resulted in the identification of 30 organic compounds* [Tables 7, 8, 9]. Concentrations ranged from <1 to 560,000 µg/l. Fifteen compounds were found in concentrations of 500 µg/l or greater.

Of the 30 compounds identified, 12 are priority pollutants.** Except for isophorone, priority pollutant concentrations ranged from a low of 1 µg/l to 140 µg/l. Isophorone was detected in 4 out of 7 composite samples with concentrations ranging from 2,500 to 5,100 µg/l (60 to 120 kg/day).

Industrial Effluent (Station 41)

Flow was determined based on the Palmer-Bowles flume and recorder located on the influent to the aeration basin. The influent to the basin remains constant for a given period of time. Plant operators change flow rates to the basin once or twice daily to either increase or decrease wastewater volume in the equalization tanks. Operators informed NEIC personnel when the flow rate to the aeration basin was changed so that the flow could be determined with lithium chloride. Results show that 20,000 to 26,300 m^3/day (average 23,100 m^3/day) of industrial wastewater was being treated in the basin.

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* One of the compounds, Tri-n-butyl phosphate was identified and confirmed but could not be quantified due to either interfering compounds or difficulties in correlation to the flame ionization chromatogram.

** Priority Pollutants are derived from the June 7, 1976 Natural Resources Defense Council (NRDC) vs. Russell Train (USEPA) Settlement Agreement.

Table 6

SUMMARY FIELD MEASUREMENTS AND ANALYTICAL DATA
SOUTH CHARLESTON SEWAGE TREATMENT COMPANY

Parameters (August) →	15	16	17	18	19	20	21	Average
			Station 39 INDUSTRIAL INFLUENT					
Flow								
m^3/day x 10^3	16.6	18.8	19.6	20.4	18.7	20.0	19.1	19.0
mgd	4.39	4.98	5.19	5.39	4.94	5.29	5.04	5.03
Temperature °C Range	30-35	29-34	30-33	29-33	30-34	30-33	28-34	
pH Range	3.6-11.9	2.3-11.5	7.5-10.8	4.3-11.4	2.0-12.0	1.5-11.3	6.7-11.6	
COD								
mg/l	2,000	3,100	2,100	3,100	2,600	2,300	3,700	2,700
kg/day	33,000	58,000	41,000	63,000	49,000	46,000	71,000	52,000
lb/day	73,000	130,000	91,000	140,000	110,000	100,000	160,000	110,000
TKN								
mg/l	24	24	32	30	54	32	59	36
kg/day	400	450	630	610	1,000	640	1,100	690
lb/day	880	1,000	1,400	1,300	2,200	1,400	2,500	1,500
NH_3-N								
mg/l	10.0	4.7	6.7	5.4	4.9	3.9	4.6	5.7
kg/day	170	90	130	110	92	78	88	110
lb/day	370	200	290	240	200	170	190	240
Chloride								
mg/l	520	430	320	140	260	190	220	300
kg/day	8,600	8,100	6,300	2,900	4,900	3,800	4,200	5,500
lb/day	19,000	18,000	14,000	6,300	11,000	8,400	9,300	12,000
Phenol								
µg/l	240	320	490	420	380	380	400	380
kg/day	4	6	10	9	7	8	8	7
lb/day	9	13	21	19	16	17	17	16

Table 7

NEUTRAL EXTRACTABLE ORGANICS SAMPLING DATA
SOUTH CHARLESTON SEWAGE TREATMENT COMPANY

Station No.	Station Description[1]	Chemical Name	Date (August) - Concentration in µg/l 15	16	17	18	19	20	21
39	Industrial Influent	Biphenyl	ND[2]	ND	430	170	130	180	120
		bis-(2-Ethoxyethyl)Ether	ND	ND	ND	370	ND	ND	MS[3]
		Butyl Carbitol	ND	1,800	6,900	15,000	48,000	2,700	14,000
		2,6-Di-tert-Butyl-p-Cresol	150	600	90	ND	ND	ND	ND
		2-Ethyl-1-Hexanol	ND	ND	4,100	11,000	ND	16,000	3,500
		Isophorone[4]	5,000	2,900	ND	ND	ND	5,100	2,500
		Phenyl Ether	ND	ND	710	330	250	360	210
		Pristane	ND	ND	ND	ND	ND	ND	ND
		Tri-n-Butyl Phosphate	MS	ND	ND	ND	ND	ND	ND
41	Industrial effluent	Biphenyl	ND	ND	ND	ND	ND	ND	ND
		bis-(2-Ethoxyethyl)Ether	ND	15	2	25	150	94	160
		Butyl Carbitol	ND	ND	ND	ND	ND	ND	ND
		2,6-Di-tert-Butyl-p-Cresol	ND	ND	ND	ND	ND	ND	ND
		2-Ethyl-1-Hexanol	ND	ND	ND	ND	ND	ND	ND
		Isophorone[4]	ND	ND	ND	ND	ND	ND	ND
		Phenyl Ether	ND	ND	ND	ND	ND	ND	ND
		Pristane	ND	22	8	14	18	16	28
		Tri-n-Butyl Phosphate	75	380	110	130	150	ND	ND
45	Discharge to Kanawha River (Outfall 001)	Biphenyl	ND	ND	ND	ND	ND	ND	ND
		bis-(2-Ethoxyethyl)Ether	ND	7	ND	16	140	160	170
		Butyl Carbitol	ND	ND	ND	ND	ND	ND	ND
		2,6-Di-tert-Butyl-p-Cresol	ND	ND	ND	ND	ND	ND	ND
		2-Ethyl-1-Hexanol	ND	ND	ND	ND	ND	ND	ND
		Isophorone[4]	ND	ND	ND	ND	ND	4,100	1,000
		Phenyl Ether	ND	ND	ND	ND	ND	ND	ND
		Pristane	ND	ND	ND	ND	ND	ND	ND
		Tri-n-Butyl Phosphate	41	26	110	34	MS	ND	ND
46	Primary sludge	Biphenyl							ND
		bis-(2-Ethoxyethyl)Ether							ND
		Butyl Carbitol							ND
		2,6-Di-tert-Butyl-p-Cresol							ND
		2-Ethyl-1-Hexanol							ND
		Isophorone[4]							ND
		Phenyl Ether							ND
		Pristane							5
		Tri-n-Butyl-Phosphate							ND
47	Industrial Grit	Biphenyl							120
		bis-(2-Ethoxyethyl)Ether							ND
		Butyl Carbitol							ND
		2,6-Di-tert-Butyl-p-Cresol							20
		2-Ethyl-1-Hexanol							ND
		Isophorone[4]							120
		Phenyl Ether							260
		Pristane							ND
		Tri-n-Butyl Phosphate							ND

1. Samples from station 43, Domestic effluent were also analyzed for these organic compounds. None of these compounds were detected.
2. ND means not detected by computerized mass spectrometric data analysis.
3. MS means the chemical was identified from its mass spectrum but interferring compounds or difficulties in correlation to the flame ionization chromatogram prevented quantitation.
4. Chemical is a priority pollutant (NRDC vs Train Consent decree, June 1976).

Table 8

VOLATILE ORGANICS DATA
SOUTH CHARLESTON SEWAGE TREATMENT COMPANY

Station Description	Industrial Influent (Station 39)							Industrial Effluent (Station 41)						
Date (August 1978)	15[a]	16	17	18	19	20	21	15	16	17	18	19	20	21
COMPOUND	Concentration (µg/l)													
Acrolein	ND[b]	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Benzene	29	29	18	16	24	5	21	40	ND	ND	ND	ND	60	ND
Bromodichloromethane	ND	1	1	1	3	ND	2	ND	ND	ND	ND	ND	ND	ND
Bromoform	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Carbon tetrachloride	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Chlorobenzene	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2-Chloroethylvinyl ether	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Chloroform	46	43	52	24	19	13	34	25	ND	1	2	ND	150	ND
Chlorodibromomethane	ND	ND	ND	ND	2	ND	ND	ND	ND	ND	ND	ND	ND	ND
1,2-Dichloroethane	ND	11	ND	14	ND	9	14	ND	ND	ND	1	ND	ND	ND
1,1-Dichloroethene	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
trans-1,2-Dichloro-ethene	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
1,2-Dichloropropane	ND	5	2	ND	4	ND	ND	ND	ND	ND	ND	ND	ND	ND
Ethylbenzene	7	44	21	27	94	ND	ND	ND	ND	ND	ND	ND	ND	ND
Methylene chloride	120	9	70	ND	15	ND	ND	150	16	ND	ND	ND	400	ND
1,1,2,2-Tetrachloro-ethane	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Tetrachloroethene	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Toluene	81	74	94	98	140	91	ND	ND	ND	3	ND	ND	ND	ND
1,1,1-Trichloroethane	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
1,1,2-Trichloroethane	ND	2	ND	1	3	2	1	ND	ND	ND	ND	ND	ND	ND
Trichloroethene	ND	1	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Vinyl chloride	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND

a Equal volume composite of three grab samples
b ND - none detected. Detection limit 1 µg/l for all components except acrolein, which has a detection limit of 50 µg/l.

Table 8 (Cont'd.)

VOLATILE ORGANICS DATA
SOUTH CHARLESTON SEWAGE TREATMENT COMPANY

Station Description	Domestic Effluent (Station 43)							Discharge to Kanawha River Outfall 001 (Station 45)						
Date (August 1978)	15	16	17	18	19	20	21	15	16	17	18	19	20	21
COMPOUND	Concentration (µg/l)													
Acrolein	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Benzene	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Bromodichloromethane	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Bromoform	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Carbon tetrachloride	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Chlorobenzene	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2-Chloroethylvinyl-ether	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Chloroform	4	15	11	8	46	21	15	2	7	4	3	15	ND	9
Chlorodibromomethane	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
1,2-Dichloroethane	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
1,1-Dichloroethene	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
trans-1,2-dichloro-ethene	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
1,2-Dichloropropane	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Ethylbenzene	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Methylene chloride	5	30	15	62	58	12	8	11	13	7	22	41	ND	29
1,1,2,2-Tetrachloro-ethane	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Tetrachloroethene	10	6	2	2	2	4	2	4	3	ND	ND	1	ND	ND
Toluene	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
1,1,1-Trichloroethane	1	5	ND	1	3	3	ND	ND	6	2	ND	ND	ND	ND
1,1,2-Trichloroethane	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Trichloroethene	ND	ND	ND	ND	2	ND	ND	ND	ND	ND	ND	ND	ND	ND
Vinyl chloride	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND

Table 9

DIRECT AQUEOUS INJECTION ORGANIC DATA
SOUTH CHARLESTON SEWAGE TREATMENT COMPANY
Concentration (mg/l)

Station Description	Date[a] (August)	acetone	methylethyl-ketone	acryloni-trile	styrene	isopro-panol	diethyl ketone	isobutro-nitrile	n-butanol	1-chloro-butane	ethanol	4-methyl-2-pentene-2-one	cellosolve acetate
Industrial Influent	15	74[b]	ND[d]	27	ND	20	<1	3.1	72	1	560	ND	ND
	16	280[b]	ND	28	ND	18	<1	ND	<1	ND	ND	ND	ND
	17	81	ND	66	2.9	59	ND	ND	37	ND	ND	ND	ND
	18	8.9[c]	ND	30[c]	1.9	46[c]	<1[c]	<1	28[c]	ND	ND	ND	ND[b]
	19	4.7	3.8	66	8.6	ND	ND	ND	ND	ND	85	ND	450[b]
	20	62	ND	35	ND	44	<1	1.5	ND	ND	ND	ND	ND
	21	200	ND	43	ND	28	ND	ND	31	ND	ND	ND	ND
Discharge to Kanawha River (Outfall 001)	15	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
	16	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
	17	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
	18	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
	19	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
	20	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
	21	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND

a Equal volume composite of three grab samples indicated by the sequence numbers.
b Value exceeded the known linear range for this parameter. A diluted aliquot of the sample was not analyzed.
c Value represents an average of two replicates.
d Not detected.

Data collected from the industrial effluent show that COD, BOD, TSS, TKN, NH_3, chloride and phenol concentrations, averaged 440, 15, 8, 19, 11, 340 and 0.01 mg/l, respectively, during the survey [Table 10]. COD, TKN and phenol removal efficiencies, based on loading, averaged 81, 38 and 97%, respectively.

NH_3 and chloride loads were higher in the effluent than those observed in the influent. The NH_3 increased from 110 to 260 kg/day and chloride from 5,500 to 7,900 kg/day. As previously noted, NH_3 is added to the aeration basin as a nitrogen source. This addition apparently also increased the amount of NH_3 discharged. The chloride increase is probably due to biological breakdown of organics in the treatment process.

Bacteriological analysis of the industrial effluent showed fecal coliform bacteria densities ranging from 20 to 4,900/100 ml (geometric mean of 84/100 ml) [Table 4].

Organic analyses of the industrial effluent resulted in the identification of 9 volatile and 3 neutral extractable compounds [Tables 7, 8]. The 9 volatile organic compounds, which were also identified in the industrial influent, were detected in concentrations ranging from 2 to 400 µg/l [Table 8]. The neutral extractable compounds - pristane, bis-(2-ethoxyethyl) ether and tri-n-butyl phosphate - were not detected in the plant influent. Concentrations of these compounds ranged from 2 to 380 µg/l [Table 7]. The 9 volatile organic compounds are priority pollutants.

An examination of the industrial influent and effluent flow data presents an anomaly. During the survey, the total amount of industrial wastewater received at SCSTC was 133,200 m^2/day, yet 161,700 m^3 was discharged, 21% greater. This difference, 28,500 m^3, could be due to inaccuracies in flow measurement (±10% on each device), the addition

Table 10

SUMMARY FIELD MEASUREMENTS AND ANALYTICAL DATA
SOUTH CHARLESTON SEWAGE TREATMENT COMPANY

Parameters (August) →	15	16	17	18	19	20	21	Average
			Station 41 INDUSTRIAL EFFLUENT					
Flow								
m^3/day x 10^3	20.4	25.9	20.0	26.3	20.2	24.0	24.9	23.1
mgd	5.40	6.85	5.28	6.94	5.34	6.35	6.58	6.11
Temperature °C Range	26-29	28-32	26-30	27-32	29-34	27-30	25-29	
pH Range	7.2-7.9	7.4-8.0	6.4-11.3	7.6-7.9	7.2-8.3	7.3-8.4	6.9-8.1	
BOD								
mg/l	10	12	14	16	21	17	14	15
kg/day	200	310	280	420	420	410	350	340
lb/day	450	690	620	930	940	900	770	760
COD								
mg/l	270	310	510	470	560	520	430	440
kg/day	5,500	8,000	10,000	12,000	11,000	12,000	11,000	9,900
lb/day	12,000	18,000	22,000	27,000	25,000	28,000	24,000	22,000
TSS								
mg/l	10	10	7	9	9	6	8	8
kg/day	200	260	140	240	180	140	200	190
lb/day	450	570	310	520	400	320	440	430
TKN								
mg/l	24	23	20	21	14	15	14	19
kg/day	490	600	400	550	280	360	350	430
lb/day	1,100	1,300	880	1,200	620	800	770	950
NH_3-N								
mg/l	15.1	18.0	11.7	12.7	6.4	8.3	6.8	11
kg/day	310	470	230	330	130	200	170	260
lb/day	680	1,000	520	740	290	440	370	580
Chloride								
mg/l	450	450	440	340	250	230	240	340
kg/day	9,200	12,000	8,800	8,900	5,100	5,500	6,000	7,900
lb/day	20,000	26,000	19,000	20,000	11,000	12,000	13,000	17,000
Phenol								
µg/l	12	6	12	9	9	10	9	10
kg/day	0.25	0.16	0.24	0.24	0.18	0.24	0.22	0.22
lb/day	0.54	0.34	0.53	0.53	0.40	0.53	0.49	0.48

of domestic secondary sludge flow to industrial primary clarifiers [Figure 1], and less volume of stored wastewater in the equalization tanks between the start and finish of the survey.

Domestic Effluent (Station 43)

Effluent flow was based on the influent Parshall flume data. The flume was checked by NEIC personnel and found to be recording flows within ±10% of actual. During the survey, domestic flows varied from 8,000 to 15,000 m^3/day (9,800 m^3/day, average). The highest flow observed, 15,000 m^3/day, occurred as a result of runoff from a rainstorm. Normal day weather flows reportedly average approximately 8,000 m^3/day.

BOD, COD, TSS, TKN, NH_3, and chloride and phenol concentrations averaged 35, 74, 44, 6.2, 2.8, 81 and 0.004 mg/l, respectively [Table 11]. The TSS concentrations (44 mg/l) are higher than would be expected from a well-operated secondary domestic WWTF. Visual observations during the survey showed up to 2.5 cm (1 inch) of floating solids on the Aero Accelator. Plant personnel informed NEIC that the floating solids resulted because of over-chlorination of the primary effluent; the excess chlorine killed some of the biota which floated and was discharged.

Fecal coliform bacteria densities for the survey ranged from 3,300 to 350,000/100 ml with a geometric mean of 27,000/100 ml [Table 4]. These high coliform bacteria densities show that the domestic effluent will significantly affect the quality of the combined discharge. As previously discussed, the domestic wastewater is disinfected after primary clarificaton, not after secondary treatment.

Organic analysis [Table 8] showed that the domestic effluent contained chloroform (4 to 46 µg/l); tetrachloroethene (2 to 10 µg/l),

Table 11

SUMMARY FIELD MEASUREMENTS AND ANALYTICAL DATA
SOUTH CHARLESTON SEWAGE TREATMENT COMPANY

Parameters (August) →	15	16	17	18	19	20	21	Average
			Station 43 DOMESTIC EFFLUENT					
Flow								
m^3/day x 10^3	15.0	10.0	9.0	8.7	8.3	8.6	8.1	9.7
mgd	3.96	2.66	2.37	2.30	2.20	2.28	2.15	2.56
Temperature °C Range	24-25	24-28	24-26	24-26	25-27	25-26	24-26	
pH Range	6.3-7.2	6.6-7.1	6.5-6.8	6.4-6.7	6.2-6.6	5.5-6.9	6.4-6.9	
BOD								
mg/l	26	24	36	76	30	28	23	35
kg/day	390	240	320	660	250	240	190	330
lb/day	860	530	710	1,500	550	530	410	730
COD								
mg/l	62	53	61	150	55	78	62	74
kg/day	930	530	550	1,300	460	670	500	710
lb/day	2,000	1,200	1,200	2,900	1,000	1,500	1,100	1,600
TSS								
mg/l	38	27	26	84	42	56	38	44
kg/day	570	270	230	730	350	480	310	420
lb/day	1,300	600	510	1,600	770	1,100	680	940
TKN								
mg/l	5.6	5.5	5.5	11,0	5.5	5.4	4.6	6.2
kg/day	84	55	49	96	46	47	37	59
lb/day	190	120	110	210	100	100	83	130
NH_3-N								
mg/l	3.0	3.0	2.8	3.7	3.3	2.2	1.3	2.8
kg/day	45	30	25	32	27	19	11	27
lb/day	99	67	55	71	61	42	23	60
Chloride								
mg/l	61	79	87	83	90	81	88	81
kg/day	910	800	780	720	750	700	720	770
lb/day	2,000	1,800	1,700	1,600	1,700	1,500	1,600	1,700
Phenol								
µg/l	6	3	11	6	0	0	3	4
kg/day	0.09	0.03	0.10	0.05	0	0	0.02	0.04
lb/day	0.20	0.07	0.22	0.12	0	0	0.05	0.09

1,1,1-trichloroethane (1 to 5 µg/l) and trichloroethene (2 µg/l). These volatile compounds are priority pollutants. Although the exact source of these organic compounds are unknown, they might originate at the Union Carbide Technical Center, a research facility, which reportedly discharges wastewater into the domestic sewer.

SCSTC Discharge (Outfall 001) - Station 45

During the study an average of 32,800 m^3/day (8.67 mgd) of wastewater was discharged to the Kanawha River. NEIC sampled and analyzed this discharge for all NPDES permit parameters, selected metals and organic compounds [Table 2].

The mixing of domestic and industrial effluent prior to discharge results in dampening out the high domestic TSS (44 mg/l) and industrial COD (440), NH_3 (11 mg/l) and chloride (340 mg/l) concentrations. Final effluent concentrations for these parameters averaged 24, 330, 8.4 and 260 mg/l, respectively [Table 3].

Composite samples results show that the discharge contained low concentrations of zinc (0.03 to 0.08 mg/l) and nickel (0.09 to 0.2 mg/l). The other metals* were below detectable limits. Both zinc and nickel are listed as priority pollutants.

Organic analyses of the discharge to the Kanawha River resulted in the identification of 7 compounds [Tables 7, 8, 9] with concentrations ranging from 7 to 4,100 µg/l. All 7 compounds, 4 identified by volatile and 3 by neutral extractable analyses, were previously identified in the industrial and/or domestic wastewaters. The 4 volatile organics (chloroform, methylene chloride, tetrachloroethene and 1,1,1-trichloroethane) are priority pollutants.

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* Samples were analyzed for Ni, Pb, Sn, Zn, As, Al, Cd, Cr and Cu.

Composite samples collected August 15, 18 and 21 were analyzed for carbaryl. The concentration on the first two samples was below the detection limit, <3 µg/l. The sample collected August 21 contained 21 µg/l carbaryl.

Solids

Samples of the industrial grit and primary domestic sludge filter cake were analyzed to determine the metal and organic content of the solids being landfilled. The metal results are listed below.

Metal[a]	Concentration µg/l	
	Primary Sludge	Industrial Grit
As	13	6
Al	4,500	4,100
Cr (total)	50	180
Cu	140	570
Ni	30	2,500
Pb	180	370
Zn	440	290
Hg	2.2	5.7

a The samples were also analyzed for Cd and Sn. These two metals were not detected in either sample.

As, Cr (total), Cu, Ni, Pb, Zn, and Hg are priority pollutants.

Organics data [Table 7] show that the domestic sludge contained 5 µg/g of pristane. The industrial grit contained 120 µg/g biphenyl, 20 µg/g 2,6-Di-tert-Butyl-p-Cresol, 120 µg/g isophorone, and 260 µg/g phenyl ether. Of these compounds only isophorone is a priority pollutant.

The domestic sludge and industrial grit are buried in the South Charleston landfill and Filmont landfill respectively. These landfills

Table 12

96-HOUR FLOW-THROUGH SURVIVAL DATA
SOUTH CHARLESTON SEWAGE TREATMENT COMPANY
August 1978

Time Period	% Survival Effluent Concentration (%)						
	Control (Kanawha River Water)	10	18	32	56	75	100
24-hour	100	100	100	100	100	100	100
48-hour	100	100	100	100	100	100	100
72-hour	100	100	100	100	100	100	100
96-hour	100	100	100	100	100	100	95

are not approved for receipt of solid wastes containing priority pollutants.

BIOLOGICAL STUDIES

Biomonitoring

A flow-through bioassay was conducted on Outfall 001 to determine whether the wastewater was acutely toxic to fish. Juvenile fathead minnows (Pimephales promelas Refinesque) averaging 4 cm in length were used as test organisms [Appendix E].

The final effluent discharge from Union Carbide South Charleston Sewage Treatment Company (Outfall 001) was not acutely toxic to fish. Ninety-five percent of the test fish survived a 96-hour exposure in 100% effluent [Table 12]. A five percent mortality rate for a 96-hour bioassay is insignificant and not indicative of toxicity.

Mutagen Testing

Analyses for mutagenic activity were performed on composite samples for Outfall 001 to determine if mutagens and potential carcinogens were present in the wastes. Each of three samples collected from the SCSTC effluent demonstrated the presence of mutagenic material upon activation with rat liver enzymes. Microsomal rat liver homogenates serve to convert certain substances into metabolites that are active mutagens and carcinogens. Basic extracts from each of the three samples

displayed a mutagenic activity ratio* of 2.5 or higher [Table 13] and a typical dose-response relationship when tested with Salmonella test strain TA 98 [Figure 2]. The mutagenic activity ratio is a measure of the tester strain mutation rate compared to control rates. A mutagenic activity ratio of 2.5 or greater correlates closely (≥90% probability) with inducement of cancer in laboratory animals.[2,3,4]

Extrapolation of this information to higher organisms (such as humans) is warranted because mutagens may alter genetic material (deoxyribonucleic acid) in a similar manner in other life forms. If a compound is mutagenic in any organism, it should not be exposed to the human population. Only one molecule of a mutagen is sufficient to cause a mutation that is also likely to be carcinogenic. Because genetic repair systems are not completely effective, safe doses of mutagens and carcinogens cannot be projected.[5,6]

Greatest reversion rates were obtained from the basic extract concentrate from the sample collected on 8/21/78 (mutagenic activity ratio of 6.4 at 103.3 ml equivalent sample volume). Acidic extracts from all three samples failed to satisfy requirements for positive mutagenicity. However, the results of mutagen testing of the basic extracts demonstrates obvious mutagenic activity; mutagenic and potential carcinogenic substances were being discharged from the SCSTC at Outfall 001.

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* The mutagenic activity ratio is a measure of the tester strain mutation rate compared to control rates. A mutagenic activity ratio of 2.5 or greater correlates closely (≥90% probability) with inducement of cancer in laboratory animals.[2,3,4] If the activity ratio is 2.5 or greater and a typical dose response relationship can be demonstrated between the tester strain and increasing concentrations of sample, the results are considered positive (i.e., the substance is a mutagen). The mutagenic activity ratio is defined as $(E-C)/c$ where E is the average number of mutant colonies per test with the sample added; C is the corresponding value for the control, and c is the historical control value of 40 averaged over 100 or more tests.

Table 13

MUTAGENIC ACTIVITY OF UNION CARBIDE INSTITUTE DISCHARGE
ON SALMONELLA TESTER STRAIN TA 98
SOUTH CHARLESTON SEWAGE TREATMENT COMPANY
August 15-21, 1978

Station Number Description	Sample[a] Type	Date-Time Collected	Extract pH	Volume of Sample[b] Concentrate Tested (µl)	Equivalent Volume of Sample (ml)	No. of Revertant Colonies Per Plate Control[c]	Experimental[d]	Mutagenic Activity Ratio[e]
45 South Charleston Sewage Treatment Company Effluent	Composite	8/15/78 2306	Base	250	51.7	40	158	3.0
				150	31.0		115	1.9
				100	20.7		110	1.8
				58	10.3		53	<1.0
				25	5.2		41	<1.0
				10	2.1		33	<1.0
				5	1.0		46	<1.0
				1	0.2		24	<1.0
45 South Charleston Sewage Treatment Company Effluent	Composite	8/18/78 2312	Base	500	96.7	40	240	5.0
				400	77.3		163	3.1
				300	58.0		144	2.6
				200	38.7		143	2.6
				100	19.3		100	1.5
				50	9.7		70	<1.0
				25	4.8		35	<1.0
				10	1.9		29	<1.0
				5	1.0		32	<1.0
				1	0.2		22	<1.0
45 South Charleston Sewage Treatment Company Effluent	Composite	8/21/78 2312	Base	500	103.3	40	296	6.4
				250	51.7		262	5.6
				100	20.7		157	2.9
				50	10.3		70	<1.0
				25	5.2		36	<1.0
				10	2.1		22	<1.0
				5	1.0		39	<1.0
				1	0.2			

a Composite Samples - Compositing was hourly for each 24-hour period; date and time listed is date and time that period ended.
b Rat-liver homogenate (S-9 mix) added.
c Value based on average of 30 control values.
d Average of 2 plates
e Mutagenic Activity Ratio = $(E-C)/\bar{c}$, where E is the no. of colonies/experimental plate, C is the no. of colonies/control plate and $\bar{c}$ is the historical control value of 40 averaged over 100 tests.

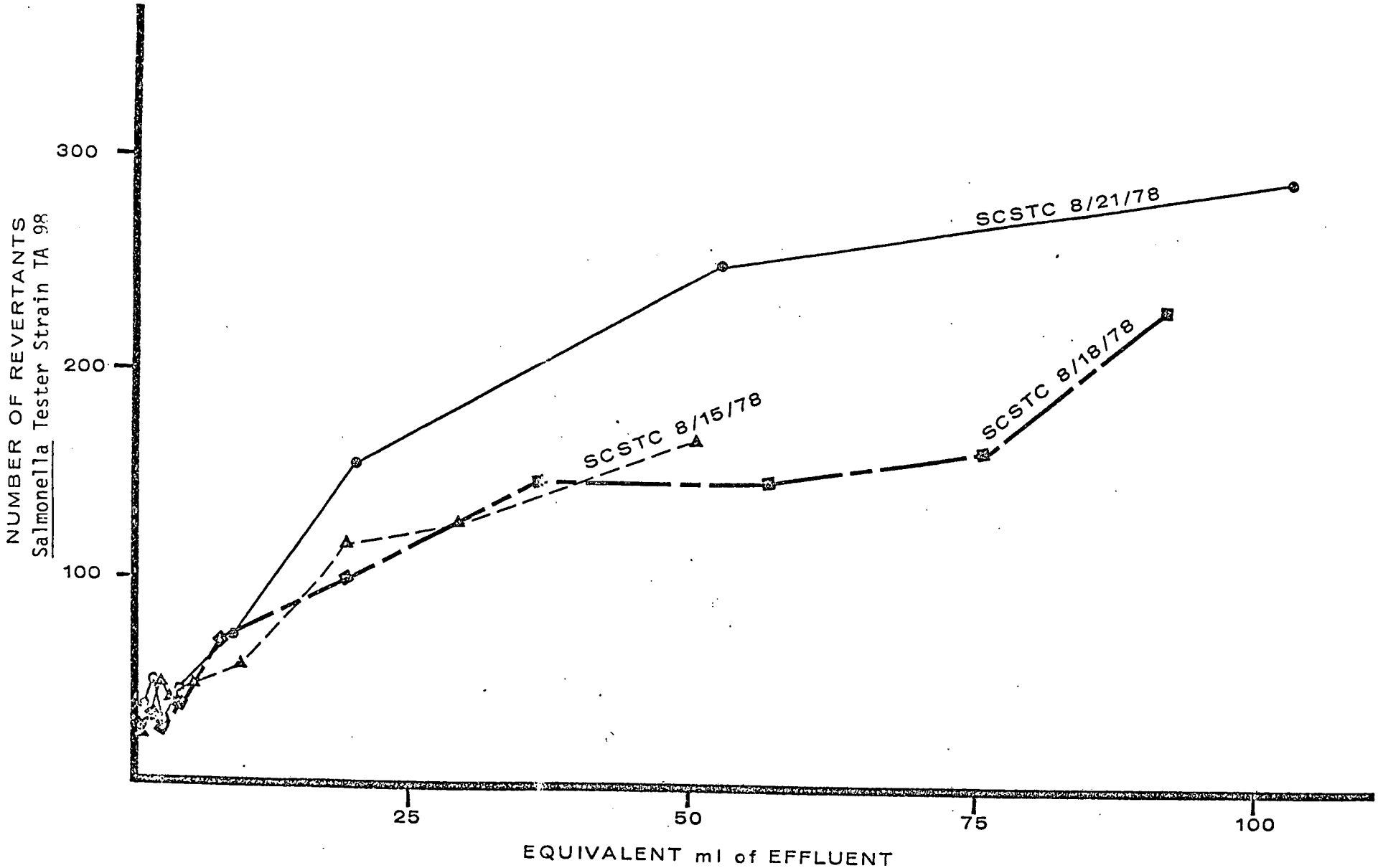

Figure 2. South Charleston Sewage Treatment Company Mutagen Testing Dose Response Curve (Basic Extract)
Salmonella Tester Strain TA 98

Data for test results that did not exhibit elevated reversion rates (negative mutagenic activity) are not presented in this report.

TOXICITY EVALUATION

A total of 34 organic compounds and 2 metals were identified in the SCSTC wastewater samples. These 36 compounds were searched in the Registry of Toxic Effects of chemical substances (RTECS)* and in the Toxline** data base to obtain health effects data [Appendix G].

THE RTECS search yielded toxicity information on 32 of the 36 compounds. The Toxline search located 578 references to health effects (animal or human) from 32 of the 36 confirmed compounds. No information on toxic effects was discovered for tri-n-butyl phosphate, bis-(2-ethoxyethyl) ether, bromodichloromethane, and chlorodibromomethane. Information on each of the other compounds is summarized in Table 14. Fifteen of the 36 compounds identified in RTECS are listed as priority pollutants.

The 36 compounds were detected in concentrations ranging from <1 µg/l to 560 mg/l. Ten of these compounds were discharged to the Kanawha River with concentrations ranging from 7 to 4,100 µg/l. The information presented in Table 14 shows that 24 compounds have demonstrated human effects associated with them. The hazards of injecting minute quantities of these organic pollutants in drinking water over long periods of time are difficult to evaluate. From the standpoint of adverse health effects, 6 of the compounds are known carcinogens, benzene to humans and carbaryl, chloroform, ethanol, trichloroethene and nickel to animals.

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* This Registry is compiled annually by the National Institute for Occupational Safety and Health.

** Toxline is a computerized bibliographic retrieval system for toxicology.

Table 14

TOXICITY OF ORGANIC COMPOUNDS
SOUTH CHARLESTON SEWAGE TREATMENT CO.

Compound Name	Molecular Formula	Chemical Abstracts Service No.	Aquatic Toxicity[a]	Other Toxicity Data[b] Route of Entry	Species	Type of Dose	Duration	Effects[e]	Exposure Limits[c]
Acetone	C_3H_6O	67-64-1	TLm 96:Over 1000 ppm	Oral	human	LDLo: 50 mg/kg			OSHA std (air): TWA 1000 ppm
				Inhalation	human	TCLo: 500 ppm		Eye	
				Inhalation	man	TCLo: 12,000 ppm	4H	Central Nerv. System	
				Oral	rat	LD50: 9,750 mg/kg			
				Inhalation	rat	LCLo: 64,000 ppm	4H		
				Inhalation	mouse	LCLo: 110,000 mg/m^3	62M		
				Intraperitoneal	mouse	LD50: 1,297 mg/kg			
				Oral	dog	LDLo: 24 g/kg			
				Intraperitoneal	dog	LDLo: 8 g/kg			
				Subcutaneous	dog	LDLo: 5 g/kg			
				Oral	rabbit	LD50: 5,300 mg/kg			
				Skin	rabbit	LD50: 20 gm/kg			
				Subcutaneous	guinea pig	LDLo: 5,000 mg/kg			
Acrylonitrile	C_3H_3N	107-13-1[d]	TLm96:100-10 ppm	Oral	human	LDLo:50 mg/kg			OSHA std (air): TWA 20 ppm (skin)
				Oral	rat	LD50:82 mg/kg			
				Oral	rat	TDLo:1,700 mg/kg	37WC	Neoplastic	
				Inhalation	rat	LCLo:500 ppm	4H		
				Inhalation	rat	TCLo:80 ppm	6H/52W	Neoplastic	
				Subcutaneous	rat	LD50:96 mg/kg			
				Parenteral	rat	LDLo:200 mg/kg			
				Oral	mouse	LD50:27 mg/kg			
				Inhalation	mouse	LCLo:900 mg/m^3	60M		
				Intraperitoneal	mouse	LDLo:10 mg/kg			
				Subcutaneous	mouse	LD50:35 mg/kg			
				Inhalation	dog	LCLo:110 ppm	4H		
				Oral	rabbit	LD50:93 mg/kg			
				Inhalation	rabbit	LCLo:258 ppm	4H		
				Skin	rabbit	LD50:280 mg/kg			
				Oral	guinea pig	LD50:50 mg/kg			
				Inhalation	guinea pig	LC50:576 ppm	4H		
Benzene	C_6H_6	71-43-2[d]	TLm96:100-10 ppm	Oral	human	LDLo:50 mg/kg			OSHA std (air): TWA 10 ppm; C1 25 Pk 50/10M/8H
				Inhalation	human	LDLo:20,000 ppm	5M		
				Inhalation	human	TCLo:210 ppm		Blood	
				Inhalation	man	TCLo:2,100 mg/m^3	4YI	Carcino-genic	
				Oral	rat	LD50:3,800 mg/kg			
				Inhalation	rat	LC50:10,000 ppm	7H		
				Intraperitoneal	rat	LDLo:1,150 mg/kg			
				Oral	mouse	LD50:4,700 mg/kg			
				Inhalation	mouse	LC50:9,980 ppm			
				Skin	mouse	TDLo:1,200 gm/kg	49WI	Neoplas-tic	

Table 14 (Cont'd.)

TOXICITY OF ORGANIC COMPOUNDS
SOUTH CHARLESTON SEWAGE TREATMENT CO.

Compound Name	Molecular Formula	Chemical Abstracts Service No.	Aquatic Toxicity[a]	Other Toxicity Data[b] Route of Entry	Species	Type of Dose	Duration	Effects[e]	Exposure Limits[c]
				Intraperitoneal	mouse	LD50:468 mg/kg			
				Subcutaneous	mouse	TDLo:2,700 mg/kg	13D (preg)	Teratogenic	
				Oral	dog	LDLo:2,000 mg/kg			
				Inhalation	dog	LCLo:146,000 mg/m^3			
				Inhalation	cat	LCLo:170,000 mg/m^3			
				Intraperitoneal	guinea pig	LDLo:527 mg/kg			
				Subcutaneous	frog	LDLo:1,400 mg/kg			
				Inhalation	mammal	LCLo:20,000 ppm	5M		
Benzene, Ethyl-	C_8H_{10}	100-41-4[d]	TLm96:100-10 ppm	Inhalation	human	TCLo:100 ppm	8H	Irritant	OSHA std (air): TWA 100 ppm (skin)
				Oral	rat	LD50:3,500 mg/kg			
				Inhalation	rat	LCLo:4,000 ppm	4H		
				Skin	rabbit	LD50:5,000 mg/kg			
				Inhalation	guinea pig	LCLo:10,000 ppm			
Biphenyl	$C_{12}H_{10}$	92-52-4		Inhalation	human	TDLo 4,400 µg/m^3		Irritant	TLV (air): 0.2 ppm
				Oral	rat	LD50: 3,280 mg/kg			
				Subcutaneous	mouse	TDLo: 46 mg/kg		Neoplastic	OSHA std (air): TWA 0.2 ppm
				Oral	rabbit	LD50: 2,410 mg/kg			
Butane, 1-chloro- (1-chlorobutane)	C_4H_9Cl	109-69-3		Oral	human	LDLo:500 mg/kg			
				Oral	rat	LD50:2,670 mg/kg			
				Inhalation	rat	LCLo:8,000 ppm	4H		
2-butanone (methyl ethyl ketone)	C_4H_8O	78-93-3	TLm96:over 1,000 ppm	Oral	human	LDLo:500 mg/kg			TLV(air):200 ppm
				Inhalation	human	TCLo:100ppm	5M	Irritant	
				Oral	rat	LD50:3,400 mg/kg			OSHA std (air): TWA 200 ppm
				Inhalation	rat	LCLo:2,000 ppm	4H		
				Inhalation	rat	TCLo:1,000 ppm	6-15D (preg)	Teratogenic	
				Intraperitoneal	mouse	LD50:616 mg/kg			
				Skin	rabbit	LD50:13 gm/kg			
Butyl alcohol (n-butanol)	$C_4H_{10}O$	71-36-3	TLm96:over 1,000 ppm	Oral	human	LDLo: 500 mg/kg			TLV(air):50 ppm (skin)
				Inhalation	human	TCLo: 25 ppm		Irritant	
				Oral	rat	LD50: 790 mg/kg			OSHA std (air): TWA 100 ppm
				Intraperitoneal	rat	LDLo: 970 mg/kg			
				Oral	mouse	LDLo: 3,000 mg/kg			
				Oral	rabbit	LDLo: 4,250 mg/kg			
				Skin	rabbit	LD50: 4,200 mg/kg			

Table 14 (Cont'd.)

TOXICITY OF ORGANIC COMPOUNDS
SOUTH CHARLESTON SEWAGE TREATMENT CO.

Compound Name	Molecular Formula	Chemical Abstracts Service No.	Aquatic Toxicity[a]	Other Toxicity Data[b] Route of Entry	Species	Type of Dose	Duration	Effects[e]	Exposure Limits[c]
Butyl Carbitol (Ethanol, 2-2-butoxy ethoxy)-	$C_8H_{18}O_3$	112-34-5	TLm96:100-10 ppm	Oral	human	LDLo:500 mg/kg			
				Oral	rat	LD50:6,560 mg/kg			
				Intraperitoneal	mouse	LD50:850 mg/kg			
				Skin	rabbit	LD50:4,120 mg/kg			
				Oral	guinea pig	LD50:2,000 mg/kg			
Carbaryl	$C_{12}H_{11}O_2N$	63-25-2	TLm96:10-1 ppm	Oral	man	TDLo: 2,800 µg/kg		Central Nerv. Syst.	OSHA std (air): TWA 5 mg/m^3
				Oral	human	LDLo: 50 mg/kg			
				Oral	rat	LD50: 400 mg/kg			
				Oral	rat	TDLo: 5,700 mg/kg	95 WI	Carcinogenic	
				Inhalation	rat	LC50: 721 mg/kg			NIOSH recm std (air):
				Oral	rat	TDLo: 50 mg/kg	(9 or 10D preg)	Teratogenic	TWA 5 mg/m^3
				Intraperitoneal	rat	LD50: 48 mg/kg			
				Implant	rat	TDLo: 80 mg/kg		Carcinogenic	
				Unknown	rat	LD50: 500 mg/kg			
				Oral	mouse	LD50: 438 mg/kg			
				Intraperitoneal	mouse	LD50: 396 mg/kg			
				Oral	dog	TDLo: 388 mg/kg	(preg)	Teratogenic	
				Oral	rabbit	LD50: 710 mg/kg			
				Oral	guinea pig	LD50: 280 mg/kg			
				Oral	guinea pig	TDLo: 300 mg/kg	(preg)	Teratogenic	
				Oral	hamster	LDLo: 250 mg/kg			
				Oral	chicken	LD50: 197 mg/kg			
				Oral	wild bird	LD50: 56 mg/kg			
Chloroform (Trichloromethane)	$CHCl_3$	67-66-3[d]	TLm96:100-10 ppm	Oral	human	LDLo:140 mg/kg			OSHA std (air):
				Inhalation	human	TDLo:1,000 mg/m^3	1Y	Systemic	TWA 50 ppm
				Inhalation	human	TCLo:5,000 mg/m^3	7M	Central Nervous System	
				Oral	rat	LD50:800 mg/kg			
				Oral	rat	TDLo:70 gm/kg	78WI	Neoplastic	NIOSH recm std (air): Cl 2 ppm/60M
				Inhalation	rat	LCLo:8,000 ppm	4H		
				Inhalation	rat	TCLo:100 ppm	7H (6-15 D preg)	Teratogenic	
				Oral	mouse	LDLo:2,400 mg/kg			

Table 14 (Cont'd.)

TOXICITY OF ORGANIC COMPOUNDS
SOUTH CHARLESTON SEWAGE TREATMENT CO.

Compound Name	Molecular Formula	Chemical Abstracts Service No.	Aquatic Toxicity[a]	Other Toxicity Data[b] Route of Entry	Species	Type of Dose	Duration	Effects[e]	Exposure Limits[c]
				Oral	mouse	TDLo:18 gm/kg	120DI	Carcinogenic	
				Inhalation	mouse	LC50:28 gm/m^3			
				Intraperitoneal	mouse	LD50:1,671 mg/kg			
				Subcutaneous	mouse	LD50:704 mg/kg			
				Oral	dog	LDLo:1,000 mg/kg			
				Inhalation	dog	LC50:100 gm/m^3			
				Intraperitoneal	dog	LD50:1,000 mg/kg			
				Intravenous	dog	LDLo:75 mg/kg			
				Inhalation	cat	LCLo:35,000 mg/m^3	4H		
				Oral	rabbit	LDLo:500 mg/kg			
				Inhalation	rabbit	LC50:59 gm/m^3			
				Subcutaneous	rabbit	LDLo:3,000 mg/kg			
				Inhalation	guinea pig	LCLo:20,000 ppm	2H		
				Inhalation	frog	LCLo:6,000 mg/m^3			
				Inhalation	mammal	LCLo:25,000 ppm	5M		
p-creosol,2,6-di-tert-butyl-	$C_{15}H_{24}O$	128-37-0		Oral	rat	LD50:2,450 mg/kg			TLV (air): 10 mg/m^3
				Oral	mouse	LD50:1,040 mg/kg			
				Intraperitoneal	mouse	LDLo:250 mg/kg			
				Oral	cat	LDLo:940 mg/kg			
				Oral	rabbit	LDLo:2,100 mg/kg			
				Oral	guinea pig	LD50:10,700 mg/kg			
2-cyclohexen-1-one, 3,5,5-trimethyl- (isophorone)	$C_9H_{14}O$	78-59-1[d]		Inhalation	human	TCLo:25 ppm		Irritant	OSHA std (air): TWA 25 ppm
				Oral	rat	LD50:2,330 mg/kg			
				Inhalation	rat	LDLo:1,840 ppm	4H		
				Skin	rabbit	LD50:1,500 mg/kg			
Ethane, 1,2-Dichloro- (Ethylene Dichloride)	$C_2H_4Cl_2$	107-06-2[d]	TLm96:1,000-100 ppm	Inhalation	human	TCLo:4,000 ppm	H	Central Nervous System	OSHA std (air): TWA 50 ppm Cl 100; Pk 200/5M/3H
				Oral	human	TDLo:428 mg/kg			
				Oral	man	LDLo:810 mg/kg			
				Oral	human	LDLo:500 mg/kg			
				Oral	rat	LD50:680 mg/kg			
				Inhalation	rat	LCLo:1,000 ppm	4H		
				Intraperitoneal	rat	LDLo:600 mg/kg			
				Subcutaneous	rat	LDLo:500 mg/kg			
				Oral	mouse	LDLo:600 mg/kg			NIOSH recm std (air): TWA 5 ppm; Cl 15
				Inhalation	mouse	LCLo:5,000 mg/m^3	2H		
				Intraperitoneal	mouse	LDLo:250 mg/kg			
				Subcutaneous	mouse	LDLo:380 mg/kg			
				Oral	dog	LDLo:2,000 mg/kg			

Table 14 (Cont'd.)

TOXICITY OF ORGANIC COMPOUNDS
SOUTH CHARLESTON SEWAGE TREATMENT CO.

Compound Name	Molecular Formula	Chemical Abstracts Service No.	Aquatic Toxicity[a]	Other Toxicity Data[b] Route of Entry	Species	Type of Dose	Duration	Effects[e]	Exposure Limits[c]
				Intravenous	dog	LDLo:175 mg/kg			
				Oral	rabbit	LD50:860 mg/kg			
				Inhalation	rabbit	LCLo:3,000 ppm	7H		
				Subcutaneous	rabbit	LDLo:1,200 mg/kg			
				Inhalation	pig	LCLo:3,000 ppm	7H		
				Inhalation	guinea pig	LCLo:1,500 ppm	7H		
				Intraperitoneal	guinea pig	LDLo:600 mg/kg			
Ethane, 1,1,1-Trichloro- (Methyl Chloroform)	$C_2H_3Cl_3$	71-55-6[d]	TLm96:100-10 ppm	Oral	human	LDLo:500 mg/kg			
				Inhalation	man	LCLo:27 gm/m^3	10M		OSHA std (air): TWA 350 ppm
				Inhalation	man	TCLo:350 ppm		Psychotropic	
				Inhalation	human	TCLo:920 ppm	70M	Central Nervous System	NIOSH recm std (air): Cl 350 ppm/15M
				Oral	rat	LD50:14,300 mg/kg			
				Inhalation	rat	LCLo:1,000 ppm			
				Inhalation	mouse	LCLo:11,000 ppm	2H		
				Intraperitoneal	mouse	LD50:4,700 mg/kg			
				Oral	dog	LD50:750 mg/kg			
				Intraperitoneal	dog	LD50:3,100 mg/kg			
				Intravenous	dog	LDLo:95 mg/kg			
				Oral	rabbit	LD50:5,660 mg/kg			
				Subcutaneous	rabbit	LD50:500 mg/kg			
				Oral	guinea pig	LD50:9,470 mg/kg			
Ethane, 1,1,2-Trichloro-	$C_2H_3Cl_3$	79-00-5[d]	TLm:96:100-10ppm	Oral	human	LDLo:50 mg/kg			OSHA std (air): TWA 10 ppm (skin)
				Oral	rat	LD50:1,140 mg/gk			
				Inhalation	rat	LCLo:500 ppm	8H		
				Intraperitoneal	mouse	DL50:994 mg/kg			
				Subcutaneous	mouse	LD50:227 mg/kg			
				Oral	dog	LDLo:500 mg/kg			
				Intraperitoneal	dog	LDLo:450 mg/kg			
				Intraveneous	dog	LDLo:95 mg/kg			
				Subcutaneous	rabbit	LDLo:500 mg/kg			
Ethanol, 2-ethoxy-acetate (Cellosolve acetate)	$C_6H_{12}O_3$	111-15-9	TLm:96:1000-100ppm	Oral	human	LDLo:500 mg/kg			TLV (air): 100 ppm (skin)
				Oral	rat	LD50:5,100 mg/kg			
				Inhalation	rat	LCLo:1,500 ppm	8H		
				Intraperitoneal	mouse	LD50:1,420 mg/kg			OSHA std (air): TWA 100 ppm (skin)
				Skin	rabbit	LD50:10,500 mg/kg			
				Oral	guinea pig	LD50:1,910 mg/kg			

Table 14 (Cont'd.)

TOXICITY OF ORGANIC COMPOUNDS
SOUTH CHARLESTON SEWAGE TREATMENT CO.

Compound Name	Molecular Formula	Chemical Abstracts Service No.	Aquatic Toxicity[a]	Other Toxicity Data[b] Route of Entry	Species	Type of Dose	Duration	Effects[e]	Exposure Limits[c]
Ether, diphenyl (phenylether)	$C_{12}H_{10}O$	101-84-8		Oral	rat	LD50:3,370 mg/kg			TLV (air): 1 ppm (vapor) OSHA std (air): TWA 1 ppm
Ethyl alcohol (ethanol)	C_2H_6O	64-17-5	TLm:96 over 1,000 ppm	Oral	child	LDLo:2,000 mg/kg			TLV (air): 1000 ppm
				Oral	human	LDLo:500 mg/kg			
				Oral	man	TDLo:50 mg/kg		Gastro-intestinal	
				Oral	man	TDLo:1,430 µg/kg		Central Nervous System	OSHA std (air): TWA 1000 ppm
				Oral	rat	LD50:14 gm/kg			
				Intraperitoneal	rat	LDLo:1,225 mg/kg			
				Intravenous	rat	LD50:1,440 mg/kg			
				Oral	mouse	LD50:7,800 µg/kg			
				Oral	mouse	TDLo:340 gm/kg	57WI	Carcinogenic	
				Intraperitoneal	mouse	TDLo:7,500 mg/kg	9D (preg)	Teratogenic	
				Intravenous	mouse	LD50:1,973 mg/kg			
				Rectal	mouse	TDLo:100 gm/kg	18WI	Carcinogenic	
				Oral	dog	LDLo:5,500 mg/kg			
				Intraperitoneal	dog	LDLo:3,000 mg/kg			
				Subcutaneous	dog	LDLo:6,000 mg/kg			
				Intravenous	dog	LDLo:1,600 mg/kg			
				Oral	cat	LDLo:6,000 mg/kg			
				Intravenous	cat	LDLo:3,945 mg/kg			
				Oral	rabbit	LD50:6,300 mg/kg			
				Skin	rabbit	LDL0:20 gm/kg			
				Intravenous	rabbit	LDL0:5,000 mg/kg			
				Oral	guinea pig	LD50:5,560 mg/kg			
				Intraperitoneal	guinea pig	LDLo:4,000 mg/kg			
				Subcutaneous	frog	LDLo:7,100 mg/kg			
Ethylene, Tetrachloro- (Tetrachloroethene)	C_2Cl_4	127-18-4[d]	TLm96:100-10 ppm	Inhalation	human	TCLo:200 ppm		Systemic	OSHA std (air): TWA 100 ppm;
				Oral	human	LDLo:500 mg/kg			
				Inhalation	man	TCLo:280 ppm	2H	Eye Effects	C1200; PK 300/5M/3H
				Inhalation	man	TCLo:600 ppm	10M	Central Nervous System	NIOSH recm std (air): TWA 50 ppm; C1 100 ppm/15M
				Inhalation	rat	LCLo:4,000 ppm	4H		
				Oral	mouse	LD50:8,850 mg/kg			
				Inhalation	mouse	LCLo:23,000 mg/m^3	2H		

Table 14 (Cont'd.)

TOXICITY OF ORGANIC COMPOUNDS
SOUTH CHARLESTON SEWAGE TREATMENT CO.

Compound Name	Molecular Formula	Chemical Abstracts Service No.	Aquatic Toxicity[a]	Other Toxicity Data[b] Route of Entry	Species	Type of Dose	Duration	Effects[e]	Exposure Limits[c]
				Intraperitoneal-mouse		LD50:5,671 mg/kg			
				Oral-dog		LDLo:4,000 mg/kg			
				Intraperitoneal-dog		LD50:2,100 mg/kg			
				Intravenous-dog		LDLo:85 mg/kg			
				Oral-cat		LDLo:4,000 mg/kg			
				Oral-rabbit		LDLo:5,000 mg/kg			
				Subcutaneous-rabbit		LDLo:2,000 mg/kg			
Ethylene, Trichloro- (Trichloroethene)	C_2HCl_3	79-01-6[d]	TLm96:1000-100 ppm	Oral-human		LDLo:250 mg/kg			OSHA std (air):
				Inhalation-human		TCLo:6,900 mg/m^3	10M	Central Nervous System	TWA 100 ppm; Cl 200; PK 300/5M/20
				Inhalation-human		TCLo:160 ppm	83M	Central Nervous System	
				Inhalation-man		TCLo:110 ppm	8H	Irritant	NIOSH recm std (air): TWA 100
				Oral-rat		LD50:4,920 mg/kg			
				Inhalation-rat		LCLo:8,000 ppm	4H		150/10M ppm;
				Oral-mouse		TDLo:135 gm/kg	27WI	Carcinogenic	
				Inhalation-mouse		LC50:3,000 ppm	2H		PK 110/10m
				Intravenous-mouse		LD50:34 mg/kg			
				Oral-dog		LDLo:5,860 mg/kg			
				Intraperitoneal-dog		LD50:1,900 mg/kg			
				Intravenous-dog		LDLO:150 mg/kg			
				Subcutaneous-rabbit		LDLo:1,800 mg/kg			
				Oral-cat		LDLo:5,864 mg/kg			
				Inhalation-cat		LCLo:32,500 mg/m^3	2H		
				Inhalation-guinea pig		LCLo:37,200 ppm	40M		
1-hexanol, 2-ethyl-	$C_8H_{18}O$	104-76-7		Oral-rat		LD50:3,200 mg/kg			
				Oral-mouse		LDLo:3,200 mg/kg			
				Skin-rabbit		LD50:2,380 mg/kg			
Isopropyl Alcohol (Isopropanol)	C_3H_8O	67-63-0	TLm 96:1000-100 ppm	Inhalation-human		TCLo:400 ppm		Irritant	TLV (air): 400 ppm (skin)
				Oral-rat		LD50:5,840 mg/kg			
				Oral-mouse		LDLo:192 mg/kg			OSHA std (air): TWA 400 ppm
				Intraperitoneal-mouse		LD50:933 mg/kg			
				Subcutaneous-mouse		LDLo:6,000 mg/kg			
				Oral-dog		LD50:6,150 mg/kg			
				Intravenous-dog		LDLo:5,120 mg/kg			
				Intravenous-cat		LDLo:1,963 mg/kg			
				Oral-rabbit		LDLo:5,000 mg/kg			
				Skin-rabbit		LD50:16 mg/kg			
				Intravenous-rabbit		LDLo:8,230 mg/kg			
				Subcutaneous-mammal		LDLo:6 mg/kg			

Table 14 (Cont'd.)

TOXICITY OF ORGANIC COMPOUNDS
SOUTH CHARLESTON SEWAGE TREATMENT CO.

Compound Name	Molecular Formula	Chemical Abstracts Service No.	Aquatic Toxicity[a]	Other Toxicity Data[b] Route of Entry	Species	Type of Dose	Duration	Effects[e]	Exposure Limits[c]
Methane, Dichloro- (Methylene Chloride)	CH_2Cl_2	75-09-2[d]	TLm96:1,000-100 ppm	Inhalation	human	TCLo:500 ppm	1YI	Central Nervous System	OSHA std (air): TWA 500 ppm; Cl 1,000; Pk 2,000/5M/2H
				Oral	human	LDLo:500 mg/kg			
				Inhalation	human	TCLo:500 ppm	8H	Blood	NIOSH recm std (air): TWA 75 ppm;
				Oral	rat	LD50:945 mg/kg			
				Inhalation	mouse	LC50:14,400 ppm	7H		Pk 500/15M
				Intraperitoneal	mouse	LD50:1,500 mg/kg			
				Subcutaneous	mouse	LD50:6,460 mg/kg			
				Oral	dog	LDLo:3,000 mg/kg			
				Inhalation	dog	LCLo:20,000 ppm	7H		
				Intraperitoneal	dog	LDLo:950 mg/kg			
				Subcutaneous	dog	LDLo:2,700 mg/kg			
				Intravenous	dog	LDLo:200 mg/kg			
				Oral	rabbit	LDLo:1,900 mg/kg			
				Subcutaneous	rabbit	LDLo:2,700 mg/kg			
				Inhalation	guinea pig	LCLo:5,000 ppm	2H		
Nickel	Ni	7440-02-0[d]		Inhalation	rat	TCLo:15 mg/m^3		Carcinogenic	OSHA std (air): TWA 1 mg/m^3 (skin)
				Subcutaneous	rat	TDLo:15 mg/kg	6WI	Neoplastic	
				Intramuscular	rat	LDLo:25 mg/kg			
				Intramuscular	rat	TDLo:1,000 mg/kg	17WI	Carcinogenic	
				Intrapleural	rat	TDLo:1,250 mg/kg	22 WI	Neoplastic	
				Parenteral	rat	TDLo:40 mg/kg	56WI	Carcinogenic	
				Intratracheal	rat	LDLo:12 mg/kg			
				Implant	rat	TDLo:250 mg/kg		Carcinogenic	
				Intraperitoneal	mouse	LD50:12 mg/kg			
				Intravenous	mouse	LDLo:50 mg/kg			
				Intravenous	dog	LDLo:10 mg/kg			
				Implant	rabbit	TDLo:165 mg/kg	2YI	Neoplastic	
				Oral	guinea pig	LDLo:5 mg/kg			
				Inhalation	guinea pig	TCLo:15 mg/m^3	91WI	Carcinogenic	
				Intramuscular	hamster	TDLo:208 mg/kg	22W	Carcinogenic	
Pentadecane, 2,6,10, 14,-tetramethyl- (Pristane)	$C_{19}H_{40}$	1921-70-6		Intraperitoneal	mouse	TDLo:1,300 mg/kg	13WI	Neoplastic	
3-pentanone (diethyl ketone)	$C_5H_{10}O$	96-22-0	TLm96:1000-100 ppm	Oral	rat	LD50:2,140 mg/kg			
				Inhalation	rat	LCLo:8,000 ppm	4H		
				Intraperitoneal	rat	LDLo:1,250 mg/kg			

Table 14 (Cont'd.)

TOXICITY OF ORGANIC COMPOUNDS
SOUTH CHARLESTON SEWAGE TREATMENT CO.

Compound Name	Molecular Formula	Chemical Abstracts Service No.	Aquatic Toxicity[a]	Other Toxicity Data[b] Route of Entry	Species	Type of Dose	Duration	Effects[e]	Exposure Limits[c]
Propane, 1,2-Dichloro-	$C_3H_6Cl_2$	78-87-5[d]	TLm96:100-10 ppm	Oral	human	LDLo:50 mg/kg			
				Oral	rat	LD50:1,900 mg/kg			OSHA std (air):
				Inhalation	rat	LCLo:2,000 ppm	4H		TWA 75 ppm
				Oral	mouse	LD50:860 mg/kg			
				Oral	dog	LDLo:5,000 mg/kg			
				Skin	rabbit	LD50:8,750 mg/kg			
				Oral	guinea pig	LD50:2,000 mg/kg			
Propanenitrile, 2-methyl- (Isobutyronitrile)	C_4H_7N	78-82-0		Oral	rat	LD50:102 mg/kg			
				Inhalation	rat	LCLo:1,000 ppm	4H		
				Skin	rabbit	LD50:310 mg/kg			
				Subcutaneous	rabbit	LDLo:9 mg/kg			
				Subcutaneous	frog	LDLo:4,800 mg/kg			
Styrene	C_8H_8	100-42-5	TLm96:100-10 ppm	Oral	human	LDLo:500 mg/kg			TLV (air):
				Inhalation	human	LCLo:10,000 ppm	30M		100 ppm
				Inhalation	human	TCLo:600 ppm		Irritant	OSHA std (air):
				Inhalation	human	TCLo:376 ppm		Central Nervous System	TWA 100 ppm; C1 200; PK 600/5M/3H
				Oral	rat	LD50:5,000 mg/kg			
				Inhalation	rat	LCLo:5,000 ppm	8H		
				Oral	mouse	LD50:316 mg/kg			
				Inhalation	guinea pig	LCLo:12 gm/m^3	14H		
Toluene	C_7H_8	108-88-3[d]	TLm96:100-10 ppm	Oral	human	LDLo:50 mg/kg			
				Inhalation	human	TCLo:200 ppm		Central Nervous System	OSHA std (air): TWA 200 ppm; C1 300; Pk 500/10M
				Inhalation	man	TCLo:100 ppm		Psychotropic	NIOSH recm std (air): TWA 100 ppm; C1 200/10M
				Oral	rat	LD50:5,000 mg/kg			
				Inhalation	rat	LCLo:4,000 ppm	4H		
				Intraperitoneal	rat	LDLo:800 mg/kg			
				Inhalation	mouse	LC50:5,320 ppm	8H		
				Skin	rabbit	LD50:14 gm/kg			
				Subcutaneous	frog	LDLo:920 mg/kg			
Zinc	Zn	7440-66-6[d]		Intraperitoneal	mouse	LD50:15 mg/kg			

Table 14 (Cont'd.)

TOXICITY OF ORGANIC COMPOUNDS
SOUTH CHARLESTON SEWAGE TREATMENT CO.

Abbreviations
(per Registry of Toxic Effects of Chemical
Substances - NIOSH - 1977 Edition)

Compound Name	Molecular Formula	Chemical Abstracts Service No.	Aquatic Toxicity[a]	Other Toxicity Data[b]					Exposure Limits[c]
				Route of Entry	Species	Type of Dose	Duration	Effects[e]	

a Aquatic Toxicity: TLm96 - 96-hour static or continuous flow standard protocol, in parts per millon (ppm).

b Other Toxicity Data:
- LD50 - lethal dose 50% kill
- LDLo - lowest published lethal concentration
- LC50 - lethal concentration 50% kill
- LDLo - lowest published lethal dose
- TDLo - lowest published toxic dose
- TCLo - Lowest published toxic concentration
- TD - toxic dose
- M - minute; H-hour; D-day; W-week; Y-year
- C - continuous
- I - intermittent

c Exposure Limits:
- NR - not reported
- NIOSH - National Institute for Occupational Safety and Health
- OSHA - Occupational Safety and Health Act of 1970
- TWA - time-weighted average concentration
- TLV - threshold limit value
- Cl - ceiling
- Pk - peak concentration

d This chemical has been selected for priority attention as point source water-effluent discharge toxic pollutant (NRDC vs Train consent decree).

e Blood - Blood effects; effect on all blood elements, electrolytes, pH, protein, oxygen carrying or releasing capacity.
Carcinogenic - Carcinogenic effects; producing cancer, a cellular tumor the nature of which is fatal, or is associated with the formation of secondary tumors (metastasis).
Central Nervous System - Includes effects such as headaches, tremor, drowsiness, convulsions, hypnosis, anesthesia.
Eye - Irritation, diplopia, cataracts, eye ground, blindness by affecting the eye or the optic nerve.
Gastrointentinal - diarrhea, constipation, ulceration.
Irritant - Any irritant effect on the skin, eye or mucous membrane.
Neoplastic - The production of tumors not clearly defined as carcinogenic.
Psychotropic - Exerting an effect upon the mind.
Systemic - Effects on the metabolic and excretory function of the liver or kidneys.
Teratogenic - Nontransmissible changes produced in the offspring.

REFERENCES

1. Ames, B.N., McCann, J., and Yamasaki, E., Methods for Detecting Carcinogens and Mutagens with the Salmonella/Mammalian Microsome Mutagenicity Test. Mutation Research, 31 (1975) 347-364.

2. Commoner, B., Chemical Carcinogens in the Environment, Presentation at the First Chemical Congress of the North American Continent, Mexico City, Mexico, December, 1975.

3. Commoner, B., Development of Methodology, Based on Bacterial Mutagenesis and Hyperfine Labelling, For the Rapid Detection and Identification of Synthetic Organic Carcinogens in Environmental Samples, Research Proposal Submitted to National Science Foundation, Feb., 1976.

4. Commoner, B., Henry, J.I., Gold, J.C., Reading, M.J., Vithatil, N.J., Reliability of Bacterial Mutagenesis Techniques to Distinguish Carcinogenic and Non-carcinogenic Chemicals, Final Report to the U.S. Environmental Protection Agency, EPA-600/1-76-022, Government Printing Office, Washington, D.C., (April 1976).

5. Hollander, A., ed., Chemical Mutagens, Principles and Methods for Their Detection, Vol. 1, New York: Plenum Press, Chap. 9, p. 267, 1971.

6. Schoneich, J., Safety Evaluation Based on Microbial Assay Procedures, Mutation Research, 41 (1976) 89-94.

APPENDIX A

CHAIN-OF-CUSTODY-PROCEDURES

CHAIN-OF-CUSTODY PROCEDURES
(March 29, 1978)

Due to the evidentiary nature of samples collected during enforcement investigations, the possession of samples must be traceable from the time the samples are collected until they are introduced as evidence in legal proceedings. To maintain and document sample possession, Chain-of-Custody procedures are followed.

SAMPLE CUSTODY

A sample is under custody if:

1. It is in your actual possession, or
2. It is in your view, after being in your physical possession, or
3. It was in your physical possession and then you locked it up to prevent tampering, or
4. It is in a designated secure area.

FIELD CUSTODY PROCEDURES

1. In collecting samples for evidence, collect only that number which provides a fair representation of the media being sampled. To the extent possible, the quantity and types of samples and sample locations are determined prior to the actual field work. As few people as possible should handle samples.

2. The field sampler is personally responsible for the care and custody of the samples collected until they are transferred or properly dispatched.

3. Sample tags (see attached) shall be completed for each sample, using waterproof ink unless prohibited by weather conditions.

4. During the course and at the end of the field work, the Project Coordinator determines whether these procedures have been followed, and if additional samples are required.

TRANSFER OF CUSTODY AND SHIPMENT

1. Samples are accompanied by a Chain-of-Custody Record (see attached). When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the Record. This Record documents transfer of custody of samples from the sampler to another person, to a mobile laboratory, or to the NEIC laboratory in Denver.

2. Samples will be properly packaged for shipment and dispatched to the appropriate NEIC laboratory* for analysis, with a separate Record prepared for each laboratory (e.g., Mobile Chemistry Lab, Mobile Biology Lab(s), Denver Chemistry Lab, Denver, Biology Lab). Shipping containers will be padlocked for shipment to the Denver laboratory. The "Courier to Airport" space on the Chain-of-Custody Record shall be dated and signed.

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* See Appendix B of NEIC Policies and Procedures Manual for Safety Precautions When Accepting Samples From Outside Sources.

3. Whenever samples are split with a facility or government agency, a separate Chain-of-Custody Record is prepared for those samples and marked to indicate with whom the samples are being split.

4. All packages will be accompanied by the Chain-of-Custody Record showing identification of the contents. The original Record will accompany the shipment, and a copy will be retained by the Project Coordinator.

5. If sent by mail, the package will be registered with return receipt requested. If sent by common carrier, a Government Bill of Lading should be used. Receipts from post offices and bills of lading will be retained as part of the permanent documentation.

LABORATORY CUSTODY PROCEDURES

1. A sample custodian or a designated alternate will receive samples for the laboratory and verify that the information on the sample tags matches that on the Chain-of-Custody Record included with the shipment. The custodian signs the custody record in the appropriate space; a laboratory staff member performs this function in the field. Couriers picking up samples at the airport, post office, etc., shall sign in the appropriate space.

2. The custodian distributes samples to the appropriate analysts. The names of individuals who receive samples are recorded in internal Branch records. Laboratory personnel are responsible for the care and custody of samples from the time they receive them until they return them to the custodian. Samples received after normal working hours may be analyzed immediately or stored as appropriate.

3. Once field-sample testing and necessary quality assurance checks have been completed, the unused portion of the sample may be disposed of. All identifying tags, data sheets and laboratory records shall be retained as part of the permanent documentation. Samples forwarded to the Denver laboratory for analysis will be retained after analyses are completed. These samples may be disposed of only upon the orders of the Chief, Enforcement Specialist Office and Assistant Director for Technical Programs, and only after all tags have been removed for the permanent file.

SAMPLE TAG

Proj. Code	Station No.	Sequence No.	Mo./Day/Yr.	Time

Station Location	Comp.	Grab

ENVIRONMENTAL PROTECTION AGENCY
OFFICE OF ENFORCEMENT
NATIONAL ENFORCEMENT INVESTIGATIONS CENTER
BUILDING 53, BOX 25227, DENVER FEDERAL CENTER
DENVER, COLORADO 80225

1501

Samplers: (Signature)

obverse

Sample Type/Preservative(s)

1. General Inorganics/Ice
2. Metals/HNO_3
3. Nutrients/H_2SO_4 & Ice
4. Oil & Grease/H_2SO_4 & Ice
5. Phenolics/H_3PO_4 & $CuSO_4$ & Ice
6. Cyanide/NaOH & Ice
7. Organic Characterization/Ice
8. Volatile Organics/Ice
9. General Organics/Ice
10. Tracer/None
11. Solids - Inorganics/Ice or Freeze
12. Solids - Organics/Ice or Freeze
13. Biol. - Inorganics/Ice or Freeze
14. Biol. - Organics/Ice or Freeze
15. Source Filter/None
16. Probe Wash/None
17. Impinger Catch/None
18. Ambient Filter/None
19. Solid Adsorbant/Ice or Freeze
20. Ambient Impinger/Amb. or Ice
21. Benthos/Ethanol or Formal
22. Bacteriology/Ice
23. Plankton/Formal; $HgCl_2$; Lugol's
24. Chlorophyll/Ice or Freeze
25. Pathogenic Bacteria/Ice
26.

Remarks:

reverse

☆GPO 777-941

ENVIRONMENTAL PROTECTION AGENCY
Office of Enforcement

CHAIN OF CUSTODY RECORD

NATIONAL ENFORCEMENT INVESTIGATIONS CENTEI
Building 53, Box 25227, Denver Federal Center
Denver, Colorado 80225

Proj. No. | Project Name

SAMPLERS: *(Signature)*

STA. NO.	SEQ. NO.	DATE	TIME	STATION LOCATION	SAMPLE TYPE: 1.General Inorganics; 2. Metals; 3. Nutrients; 4. Oil & Grease; 5. Phenolics; 6. Cyanide; 7. Organic Characterization; 8. Volatile Organics; 9. General Organics; 10. Tracer; 11. Solids - Inorganics; 12. Solids - Organics; 13. Biol - Inorganics; 14. Biol - Organics; 15. Source Filter; 16. Probe Wash; 17. Impinger Catch; 18. Ambient Filter; 19. Solid Adsorbant; 20. Ambient Impinger; 21. Benthos; 22. Bacteriology; 23. Plankton; 24. Chlorophyll; 25. Pathogenic Bacteria; 26.; 27.; 28.	NUMBER OF CONTAINERS	Remarks
				TOTAL NO. OF CONTAINERS			

Relinquished by: *(Signature)*	Date/Time	Received by: *(Signature)*	Relinquished by: *(Signature)*	Date/Time	Received by: *(Signature)*
Relinquished by: *(Signature)*	Date/Time	Received by: *(Signature)*	Relinquished by: *(Signature)*	Date/Time	Received by Courier *(Signature)*
Relinquished by Courier *(Signature)*	Date/Time	Received by Mobile Lab *(Signature)*	Relinquished by Mobile Lab *(Signature)*	Date/Time	Received by Courier *(Signature)*
Method of Shipment:		Shipped by: *(Signature)*	Courier from Airport *(Signature)*	Received for Laboratory by: *(Signature)*	Date/Time

Distribution: Original Accompanies Shipment; Copy to Coordinator Field Files

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APPENDIX B

LITHIUM FLOW VERIFICATION PROCEDURES

AND SAMPLING PROCEDURES

LITHIUM FLOW VERIFICATION PROCEDURES

Flow verification was accomplished with the tracer dilution technique, using lithium as the tracer. The concept employed is that mass is conserved (i.e., mass of tracer in equals mass of tracer out). Fundamental to the use of this technique are the following conditions:

1. A conservative tracer.
2. A constant tracer injection rate and an accurate measurement of the rate.
3. An accurate measurement of the tracer concentrate, background tracer levels, and diluted tracer in the flow stream to be measured.
4. Complete mixing in the flow stream to be measured.

It was determined that all these respective criteria could be met by:

1. Using lithium (Li) in the form of lithium chloride as a tracer. Previous studies have shown that spiking various types of wastewater with known amounts of lithium results in an overall average recovery of 100%.
2. Metering the injected tracer solution with low flow rate, high precision pumps. During verification, injection rate was checked at least twice with a graduated cylinder and stop watch.

3. Measuring Li concentration with a Perkin-Elmer Model 403 Atomic Absorption Spectrophotometer. This instrument was calibrated before each use with lithium standards of known concentration. Concentrate samples were analyzed each time a batch was mixed. Background samples were collected and analyzed each time a flow measurement was performed.
4. Injecting the lithium chloride concentrate solution into the suction side of the effluent pump and monitoring the diluted Li tracer on the discharge side.

Flow was calculated with the following equation:

$$Q = \frac{q\ Cq\ F}{C-C_b}$$

where Q is unknown flow (mgd)

q is injection rate (l/min)

Cq is lithium concentration of injection solution (mg/l)

C is lithium concentration downstream of injection (mg/l)

C_b is background concentration of lithium (mg/l)

F is factor to convert l/min to mgd

$(380.45 \times 10^{-6} \frac{\text{min - gal}}{\text{day-liter}})$

SAMPLING PROCEDURES

Composite samples were collected by hand at regular intervals throughout a 24-hour period and aliquoted proportional to the volume of the discharge into iced sample containers. For those samples whose nature could change during the collection period chemical preservatives were added to the sample container prior to the start of the collection period. Each of the sample aliquots were chemically preserved upon collection. At the end of the sampling period, the chemically unpreserved portion of the sample was transferred into appropriately preserved containers, identified and transported to either NEIC mobile laboratories located at the South Charleston Sewage Treatment Company plant or the NEIC laboratory in Denver, Colorado.

Grab samples were handled as discussed above with the exception that the sample consisted of a single aliquot rather than multiple samplings.

APPENDIX C

ANALYTICAL METHODS AND QUALITY CONTROL

CHEMISTRY ANALYTICAL METHODOLOGY AND QUALITY CONTROL

The analytical procedures used by the Chemistry Branch are described in the following sections which are organized by working groups Inorganics, Organics, and Trace Metals. The quality control procedures and data used to verify the quality of the analytical data are also discussed.

INORGANICS

The samples from this study were analyzed for the following inorganic parameters - BOD, TSS, COD, NH_3, total Kjeldahl nitrogen, chloride and phenolics. Methods approved by the EPA for the NPDES program (40 CFR 136, Federal Register, December 1, 1976) were used to analyze all samples. The references to the methods for each parameter are listed in Table 1 below.

Parameter	Technique	Detection Limit, mg/l	Reference
BOD	Multiple bottle dilution	2	Std. Methods, pg. 543
COD	Dichromate reflux titration	5	Std. Methods, pg. 550
TSS	Glass fiber filter filtration	1	Std. Methods, pg. 94
NH_3	Automated phenolate	0.05	Std. Methods, pg. 616
Phenolics	4-AAP chlorimetric	0.001	Std. Methods, pg. 574
TKN	Kjeldahl digestion,	0.2	EPA Manual, pg. 175
	Automated phenolate		Std. Methods, pg. 616
Chloride	Mercuric nitrate	1	Std. Methods, pg. 304

Std. Methods - "Standard Methods for the Examination of Water and Wastewater", 14th edition (1975).
EPA Manual - "Methods for Chemical Analysis of Water and Wastes", 1974.

Written methods prepared from "Standard Methods" for BOD and TSS are included as Attachments I & II. Additional precautions taken during the analysis of the samples are discussed below by parameter.

BOD

The dissolved oxygen meter was calibrated by the azide modification of the Winkler method ("Standard Methods", 14 edition, 1975, pg 443) to assure accurate D.O. Measurements. Samples were seeded with seed material that was acclimated to the specific waste being studied. The D.O. depletions were normal for all dilutions of all samples.

Quality control consisted of duplicate analysis of seven samples and analysis of EPA reference sample #276-2 on six different days. Additional quality control procedures are described in Attachment I. Since two duplicate samples did not have valid dilutions, the precision was calculated from five sets of data. The relative standard deviation of the duplicate results is 25%. One reference sample result was invalid because of improper preparation. The mean accuracy of the five valid reference sample results is 94.5%.

TSS

The analytical and quality control procedures described in Attachment II were closely followed. The relative standard deviation of five duplicate determinations is 3%. The mean accuracy of analysis of a standard reference sample on four different days was 105%.

COD

All samples above and below 100 mg/l were analyzed using the high and low level reagents, respectively. Four samples were analyzed in duplicate with a mean RSD of 0.4%. Three samples were spiked with a mean recovery of 107%. Two reference samples were analyzed with a mean accuracy of 98%.

Chloride

Low and high level mercuric nitrate reagents were used for samples below and above 25 mg/l. Eight samples were spiked with a mean recovery of 100%. A reference sample was analyzed on five days with an accuracy of 100%. Fifteen samples were analyzed in duplicate with a mean FSD of 1%.

Ammonia

Two auto-analyzer method was adapted to 0-30 mg/l full scale by adding a dilution loop onto the front end of the manifold. Two reference samples were analyzed six times each with accuracies of 98 and 104%. Seven samples were analyzed in duplicate with five samples below the detection limit. The RSD of the two pairs of data is 1.6%.

Phenolics

All absorbances were measured against a chloroform blank. Three samples were spiked with a mean recovery of 98%. One reference sample was analyzed with 92% accuracy.

TKN

The method was set up for 20 mg/l TKN-N full scale. Samples over 20 mg/l were diluted and re-digested before analysis. A reference sample was analyzed five times with 92% accuracy.

Metals

The samples from this study were analyzed for the following metals: Al, As, Cd, Cr, Cu, Ni, Pb, Sn, and Zn. The samples consisted of water samples, a primary domestic sludge sample, and an industrial grit sample. Methods approved by the EPA for the NPDES program (40 CFR 136, Federal Register, December 1, 1976) were used in the analysis of all water samples. The preparation techniques for the domestic sludge sample and the industrial grit sample for all metals except arsenic are based on that described in the Chemistry Laboratory Manual - Bottom Sediments of the Great Lakes Region Committee on Analytical Methods, 1969. The preparation and analysis techniques for the domestic sludge sample and the industrial grit sample for arsenic were performed using approved methods listed in the Federal Register of December 1, 1976 (40 CFR 136). The references to the methods used in the analysis of the water samples for each metal and the detection limits for each metal are listed in Table 1. The references to the methods used in the analysis of the primary domestic sludge sample and the industrial grit sample for each metal and the detection limits for each metal are listed in Table 2. The detection limits in Table 1 for the water samples are reported in units of milligrams per liter. The detection limits in Table 2 for the primary domestic sludge sample and the industrial grit sample are reported in units of micrograms per gram. The detection limits in Table 2 assume a one gram dry weight of sediment and a 100 ml digestion volume.

The methods listed in Tables 1 and 2 for each element were closely followed. There were no significant deviations from the approved methods. As an added precaution, all analyses were performed using background correction procedures in order to preclude extraneous signals from the sample matrix.

Water Samples

Aluminum: Sample replicates and spikes were analyzed for aluminum. Only one sample replicate contained a detectable quantity of aluminum. This replicate agreed with the original sample within 17%. The recoveries for the sample spikes ranged from 80% to 100% with an average recovery of 87%. This represents a slight negative bias in the aluminum results. The EPA reference standard #3, lot 575, was analyzed. The experimental value was 0.9 mg/l while the true value was 0.904 mg/l aluminum.

Arsenic: Sample replicates and spikes were analyzed for arsenic. Only one sample replicate contained a detectable quantity of arsenic. This replicate agreed with the original sample within 12%. The recoveries for the sample spikes ranged from 110% to 150%, with an average

recovery of 130%. This represents a positive bias in the arsenic results. The EPA reference standards #2 and #3, lot 575, were analyzed. The experimental values were 0.11 and 0.16 mg/L, while the true values wre 0.109 and 0.154 mg/l arsenic respectively.

Cadmium: Sample replicates and spikes were analyzed for cadmium. None of the sample replicates contained detectable quantitites of cadmium. The recoveries for the sample spikes ranged from 104 to 110%, with an average recovery of 106%. The EPA reference standard #3, lot 575, was analyzed. The experimental value was 0.06 mg/L, while the true value was 0.073 mg/l cadmium.

Chromium: Sample replicates and spikes were analyzed for chromium. None of the sample replicates contained detectable quantities of chromium. The recoveries for the sample spikes ranged from 102% to 104% with an average recovery of 103%. The EPA reference standard #3, lot 575, was analyzed. The experimental value was 0.2 mg/l, while the true value was 0.204 mg/l chromium.

Copper: Sample replicates and spikes were analyzed for copper. Only one sample replicate contained a detectable quantity of copper. This replicate agreed with the original sample within 26%. This represents a difference in concentration of only 0.03 mg/l. The recoveries for the sample spikes ranged from 96% to 104% with an average recovery of 99%. The EPA reference standard #3, lot 575, was analyzed. The experimental value was 0.1 mg/l, while the true value was 0.102 mg/l copper.

Nickel: Sample replicates and spikes were analyzed for nickel. The replicate results varied from 3% to 35% relative percent difference. The 35% difference represents a concentration difference of only 0.03 mg/l. The recoveries for the sample spikes ranged from 102 to 110% with an average recovery of 107%. The EPA reference standard #3, lot 575, was analyzed. The experimental value was 0.21 mg/l, while the true value was 0.152 mg/l nickel.

Lead: Sample replicates and spikes were analyzed for lead. None of the sample replicates contained detectable quantities of lead. The recoveries for the sample spikes ranged from 92% to 134% with an average recovery of 113%. This represents a slight positive bias in the lead results. The EPA reference standard #3, lot 575, was analyzed. The experimental value was 0.45 mg/l, while the true value was 0.352 mg/l lead.

Tin: Sample replicates and spikes were analyzed for tin. None of the sample replicates contained detectable quantities of tin. The recoveries for the sample spikes ranged from 58 to 90% with a mean recovery of 74%. This represents a negative bias in the determination of tin. This is not surprising since tin is known to be unstable in solution. The EPA reference standard #3, lot 575, does not contain tin. Therefore, no AQC data is available for tin.

Zinc: Sample replicates and spikes were analyzed for zinc. The relative percent difference for the zinc replicates ranged from 0% to 29%. The 29% relative percent difference represents a concentration difference of only 0.015 mg/l. The recoveries for the sample spikes ranged from 152% to 168% with an average recovery of 159%. This represents a positive bias in the zinc results. Laboratory contamination of the zinc spikes was investigated by determining the zinc concentration of laboratory reagent blanks using the same acid that was used to preserve the samples in the field. The laboratory reagent blanks were found to contain no zinc. The EPA reference standard #3, lot 575, was analyzed for zinc. The experimental value was 0.17 mg/l, while the true value was 0.174 mg/l zinc. The fact that the experimental results for EPA reference standard #3, lot 575, were in good agreement with the true value provided by the Environmental Monitoring and Support Laboratory, Cincinnati, Ohio, together with the fact that the reagent blank contained no zinc, indicates that the water samples were inadvertently spiked at a higher level than that which was expected. The average value of the field blanks was 0.04 mg/l zinc. Sample results having this approximate concentration are questionable.

Sediment Samples

Triplicate analyses for each metal contained in each of the two sediment samples were performed.

Sludge: This sample contains all of the metals analyzed except cadmium and tin at detectable concentrations. Aluminum is present at a high level. Zinc, mercury, and lead are present at concentrations which merit attention.

Sample replicates were analyzed for all metals. The relative standard deviation for the three replicates is listed for each metal in Table 3. The relative standard deviations (RSD's) range from 5% to 29%. The high RSD for nickel is a result of the randomness introduced in sampling plus the low precision due to the relatively low nickel concentration (31 µg/g) found in the sludge sample.

Grit: This sample contains all the metals analyzed except tin and cadmium. Aluminum and nickel were found in high concentrations. Chromium, copper, mercury, lead and zinc are present at concentrations which merit attention.

Sample replicates were analyzed for all metals. The relative standard deviation for the three replicates is listed for each metal in Table 3. The relative standard deviations range from 2% to 20%.

Table 1

ANALYTICAL METHODS AND DETECTION LIMITS - WATER SAMPLES

Metal	Technique	Detection Limit, mg/l	Reference[1]
Al	Flame Atomic Absorption	0.3	A, p. 92
As	Flameless Atomic Absorption	0.002	B
Cd	Flame Atomic Absorption	0.03	A, p. 101
Cr	Flame Atomic Absorption	0.04	A, p. 105
Cu	Flame Atomic Absorption	0.04	A, p. 108
Ni	Flame Atomic Absorption	0.06	A, p. 141
Pb	Flame Atomic Absorption	0.2	A, p. 112
Sn	Flame Atomic Absorption	1.0	A, p. 150
Zn	Flame Atomic Absorption	0.01	A, p. 155

[1]A - Methods for Chemical Analysis of Water and Wastes, U.S. Environmental Protection Agency, (1974).
B - Atomic Absorption Newsletter, 14, 109 (1975).

Table 2

ANALYTICAL METHODS AND DETECTION LIMITS - PRIMARY DOMESTIC SLUDGE SAMPLE AND INDUSTRIAL GRIT SAMPLE

Element	Technique	Detection Limit, (µg/g)	Reference[1]
Al	Flame Atomic Absorption	18	A;B, p. 92
As	Flameless Atomic Absorption	0.3	C
Cd	Flame Atomic Absorption	4	A;B, p. 101
Cr	Flame Atomic Absorption	2	A;B, p. 105
Cu	Flame Atomic Absorption	3	A;B, p. 108
Hg	Flameless Atomic Absorption	0.01	A;B, p. 134
Ni	Flame Atomic Absorption	6	A;B, p. 141
Pb	Flame Atomic Absorption	55	A;B, p. 112
Sn	Flame Atomic Absorption	200	A;B, p. 150
Zn	Flame Atomic Absorption	2	A;B, p. 155

[1]A - Chemistry Laboratory Manual - Bottom Sediments, Great Lakes Region Committee on Analytical Methods, U.S. EPA, Federal Water Quality Administration, (December 1969).
B - Methods for Chemical Analysis of Water and Wastes, U.S. EPA (1974).
C - Absorption Newsletter, 14, 109 (1975).

Table 3

RELATIVE STANDARD DEVIATION (%) FOR TRIPLICATE ANALYSIS OF SLUDGE AND GRIT SAMPLES

Element	Sludge Sample	Grit Sample
Al	14	20
As	20	9
Cd	N.D.[1]	N.D.[1]
Cr	12	7
Cu	10	17
Hg	12	8
Ni	29	2
Pb	17	7
Sn	N.D.[1]	N.D.[1]
Zn	5	14

[1] N.D. - Not detectable.

ORGANICS

Several techniques for the analysis of organic compounds were utilized for the waste source evaluation, Union Carbide facilities and South Charleston WWTP Survey. Identification of individual organic compounds was made by combined gas chromatography/mass spectrometry (GC/MS) while capillary column gas chromatography (CPGC) was used for quantitation and confirmation of identity. The samples were analyzed for neutral extractables, volatiles, and selected samples were analyzed for priority pollutants. Other samples, notably nonpurgeables, were analyzed by direct aqueous injection analysis (DAI). Carbaryl was analyzed by high pressure liquid chromatography (HPLC).

NEUTRAL EXTRACTABLE ANALYSIS

GC/MS Identification: Methylene chloride extracts of the water, and acetone extracts of the sediment samples were concentrated to small volumes and exchanged with isooctane and analyzed by GC/MS. The initial identification was made using a manual search utilizing reference spectra analyzed under the same instrumental conditions used for the samples.

A library of standard spectra of the commonly occurring compounds was made using a computer assisted evaluation program.[1] In those instances where other than the commonly occurring compounds appeared, a more complete search was made utilizing the complete computer library and a follow up manual search.[2,3,4,5]

Capillary Column Gas Chromatography: All the sample extracts were analyzed by capillary column gas chromatography. Initial screening and quantitation were carried out on this gas chromatograph. Compounds were identified by coincidence of retention times with standards and quantitation was made using peak height measurement.

Packed Column Gas Chromatography: All the extracts were analyzed by packed column gas chromatography using a computer controlled automatic injector. Initial screening was carried out on this gas chromatograph.

REFERENCES

1. "INCOS Data System - MSDS Operator's Manual, Revision 3". Finnigan Instruments, March 1978.

2. "Eight Peak Index of Mass Spectra", Mass Spectrometry Data Centre, Aldemaston, Reading, UK. Second Edition 1974.

3. "Registry of Mass Spectral Data", Stenhagen, Abrahamson and McLafferty, John Wiley & Sons, New York 1974.

4. "Atlas of Mass Spectra Data" edited by: Stenhagen, Abrahamson and McLafferty, John H. Wiley & Sons, New York 1969.

5. Computer Assisted Evaluation of Organic Priority Pollutant GC/MS Data - NEIC, September 1978.

Quality Control: Quality control procedures consisted of analysis of selected duplicate samples, analysis of solvent and procedure blanks to identify interferences, and gas chromatographic analysis of standards on a daily basis to confirm the integrity of the GC system. For mass spectrometry, a daily calibration was used to tune the mass spectrometer, and assure the integrity of the complete system. The quality control procedures are documented in the attached methodologies (Attachments 5, 6, 7, 8, 9, 10).

DIRECT AQUEOUS INJECTION ANALYSIS (DAI)

Selected samples were analyzed by DAI gas chromatography/mass spectrometry (GC/MS). An aliquot of a sample is injected directly into the inlet system of a gas chromatograph interfaced to a mass spectrometer equipped with a computerized data system. Generally, low boiling semi-volatile compounds that purge poorly are analyzed by this method.

Quality Control: Blanks, duplicate and spiked samples were analyzed concurrently with the survey samples. None of the thirteen selected DAI compounds were found in any of the three blank samples.

Five spiked samples representing eleven compounds were analyzed. (One sample contained as many as three spiked compounds. Some compounds, such as acetone were spiked into more than one sample). Of the eleven discrete spikes the mean recovery was 116% with a Relative Standard Deviation of 29%.

Two sets of replicates were analyzed with four compounds detected. The average percent Relative Standard Deviation (% RSD) was 15. The average percent difference of all sets of replicates was 22.

VOLATILES ANALYSIS

GC/MS Identification: An aliquot (5 ml) of a water samples was purged with inert gas. The lower molecular weight purgable organic compounds were stripped from the sample and trapped on a porous polymer. These compounds were then desorbed from the column by reversing the gas flow and rapidly heating the trap. The volatile organics released were collected on an analytical GC column at room temperature. After collection, the GC column oven was heated at a uniform rate and the eluted compounds analyzed by the mass spectrometer. The common volatile organic solvents are all identified using this technique and it also includes the identification of the volatile priority pollutants. This procedure is the method recommended for the priority pollutants.[1] The identification again was made using a computer assisted evaluation program as for the neutral extractables.[2] A library of standard spectra was created by analyzing all the commonly occurring organics in the Kanawha samples, and adding these to the library. The samples were routinely searched for these compounds for each sample analyzed by GC/MS.

Quantitative results were obtained using an internal standard computer technique.[2,3]

REFERENCES

1. "Samples and Analysis Procedures for Screening of Industrial Effluents for Priority Pollutants", U.S. EPA, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio, March 1977, revised April 1977.

2. "INCOS Data System - MSDS Operator's Manual - Revision 3", Finnigan Instruments, March 1978.

3. Computer Assisted Evaluation of Organic Priority Pollutant GC/MS Data - NEIC, September 1978.

Quality Control: Quality control procedures consisted of daily routine calibration of the GC/MS, analysis of an organics free water blank, and a standard mix at a concentration near midpoint of the standard calibration curve. The calibration curve was previously established by analyzing each standard over a typical working range of 20 to 200 ppb concentration, with response factors calculated relative to an internal standard. Field blanks were analyzed with each set of samples. Replicate analyses were run on at least two samples for every set of twenty samples or less.

Blanks

One contaminant, methylene chloride, appeared consistently in the blank results. Blanks for the fifteen days of analysis gave a methylene chloride value of 3 ± 2 µg/l.

Summary of blank results (µg/l)

Compound	Times Detn (15 samples)	Range of Values	Average
Methylene chloride	12	2-13	3 ± 2
Toluene	2	2-5	nil
1,1,1-Trichloroethane	1	3	nil

Duplicates

Nine samples, six of them composites, were analyzed in duplicate. Ten compounds of interest were determined in these analyses. The results are summarized as follows:

Compound	Times Detn (9 samples)	Deviation
Benzene	2	± 8%
Bromodichloromethane	1	± 100%
Carbon tetrachloride	1	± 50%
Chloroform	6	± 27%
1,2-Dichloroethane	1	± 20%
Ethylbenzene	1	± 80%
Methylene chloride	6	± 45%
Tetrachloroethane	1	± 25%
Toluene	2	± 48%
1,1,1-Trichloroethane	1	± 17%

Recoveries

Four samples were spiked with standard mix to give each component at a concentration of 200 µg/l. Recoveries are listed below:

Compound	Percent Recovery
Benzene	60
Bromodichloromethane	108
Bromoform	127
Carbon tetrachloride	80
Chlorobenzene	86
2-Chloroethylvinyl ether	125
Chloroform	88
Chlorodibromomethane	113
1,2-Dichloroethane	114
1,1-Dichloroethene	81
trans-1,2-Dichloroethene	77
1,2-Dichloropropane	84
Ethylbenzene	72
Methylene chloride	93
1,1,2,2-Tetrachloroethane	140
Tetrachloroethene	83
Toluene	87
1,1,1-Trichloroethane	78
1,1,2-Trichloroethane	121
Trichloroethene	85
Vinyl chloride	97
Average	95

EPA Quality Control Sample

An internal quality control sample, prepared by the EPA Environmental Monitoring and Support Laboratory Quality Assurance Branch, Cincinnati, was analyzed in triplicate. This QC sample, containing volatile organics, was number 1276 WS.

Compound	Analytical Results ug/l	"True" Values	Error %
Bromochloromethane (IS)	180 ± 20	200	10
Bromodichloromethane	13 ± 2	12	8
Bromoform	13 ± 1	14	8
Carbon tetrachloride	9 ± 1	13	31
Chloroform	60 ± 7	68	12
Chlorodibromomethane	12 ± 1	17	29
1,2-Dichloroethane	23 ± 2	27	15
Tetrachloroethene	8 ± 1	9	11
1,1,1-Trichloroethane	9 ± 1	11	18
Trichloroethene	17 ± 2	19	11

PRIORITY POLLUTANTS ANALYSIS

GC/MS Identification: Selected samples were analyzed for priority pollutants by GC/MS using the recommended EPA procedure.[1] The volatiles were measured using the same technique described previously for the volatiles analysis, because both techniques are the same. The extractable organics were analyzed for both acids, and neutrals, and bases combined as recommended.

REFERENCES

1. "Sampling and Analysis Procedures for Screening of Industrial Effluents for Priority Pollutants", U.S. EPA, EMSL, Cincinnati, Ohio, March 1977, revised April 1977.

2. Computer Assisted Evaluation of Organic Priority Pollutant GC/MS Data - NEIC, September 1978.

ATTACHMENT I

BIOCHEMICAL OXYGEN DEMAND - DO PROBE PROCEDURE

(5 Days, 20°C) STORET NO. 00310

1. Scope and Application

1.1 The biochemical oxygen demand test is a laboratory bioassay procedure used to estimate the quantity of oxygen that is required to stabilize the biodegradable matter in a wastewater.

1.2 The test was originally designed for and works most reliably on raw and treated domestic wastes. The test can be applied to industrial wastes with careful attention to interferences and correct choice of biological seed.

2. Summary of Method

2.1 An appropriate number of dilutions of each sample are prepared using dilution water with added nutrients so that at least one dilution has a depletion of at least 2 mg/l and a residual DO of at least 1 mg/l after incubation for 5 days in the dark at 20°C.

2.2 Dissolved oxygen is measured by a DO probe based on the polarographic principle. The probe is calibrated with air saturated water at known temperature and atmospheric pressure.

3. Sample Handling and Preservation

3.1 Samples should be stored in ice or in a refrigerator at 4°C and analyzed as soon as possible but no later than 24 hours after collection.

4. Apparatus

4.1 Glass or tin-lined still to produce distilled water.

4.2 Five gallon glass bottles wrapped with nylon tape to store dilution water.

4.3 Incubation bottles, approximately 300 ml, with standard ground glass tops and plastic caps to maintain water seals. The exact volume of each bottle is measured using water at 20°C with class A volumetric glassware and any that are not 300 ± 5 ml are discarded.

4.4 An incubator with a continuous temperature recorder controlled at 20° ± 1°C. A calibrated mercury thermometer is placed in the incubator in a water-containing flask and the temperature is checked daily.

4.5 A dissolved oxygen meter, automatically temperature compensated, if possible, with a self-stirring probe.

4.6 A Tekmar SDT Tissuemizer with variable speed control to homogenize samples.

4.7 Barometer

5. Reagents

5.1 Distilled water, free of organic contaminants as indicated by the Permanganate Test as follows: Determine the consumption of potassium permanganate by adding 0.20 ml of $KMnO_4$ solution (0.316 g/l) to 500 ml of the distilled water and 1 ml of conc. H_2SO_4 in a stoppered glass bottle. The water has passed the test if the permanganate color does not disappear in less than 10 minutes upon standing at room temperature. Ideally, the color should be retained for 30 minutes.

5.2 Phosphate buffer solution: Dissolve 8.5 g potassium di-hydrogen phosphate, KH_2PO_4, 21.75 g dipotassium hydrogen phosphate, K_2HPO_4, 33.4 g disodium hydrogen phosphate heptahydrate, $Na_2HPO_4 \cdot 7H_2O$ and 1.79 g ammonium chloride NH_4Cl in about 500 ml distilled water and dilute to one liter. The pH of this buffer should be 7.2. Store in the refrigerator and discard (including any of the following reagents) if there is any sign of biological growth in the bottle.

5.3 Magnesium sulfate solution: Dissolve 22.5 g $MgSO_4 \cdot 7H_2O$ in distilled water and dilute to one liter.

5.4 Calcium chloride solution: Dissolve 27.5 g anhydrous $CaCl_2$ in distilled water and dilute to one liter.

5.5 Ferric chloride solution: Dissolve 0.25 g $FeCl_3 \cdot 6H_2O$ in distilled water and dilute to one liter.

5.6 1 N H_2SO_4 and 1 N NaOH solutions.

5.7 Sodium sulfite solution, 0.028 N: Dissolve 1.77 g anhydrous Na_2SO_3 in one liter distilled water. Prepare daily.

5.8 Reagent grade potassium iodide.

5.9 Starch indicator solution: Add a cold water suspension of 59 g soluble starch to 800 ml of boiling water, with stirring and boil for a few minutes. Cool, dilute to approximately 1 liter and let settle overnight. Use supernate and preserve with 5 ml of chloroform.

5.10 Glucose-glutamic acid solutions: A) Dissolve 150 mg of each in distilled water and dilute to 1 liter. B) Dissolve 100 mg of each in distilled water and dilute to 1 liter. Split up each solution into 25 ml bottles or tube, autoclave at 121°C for 1/2 hour, and store at 4°C or prepare fresh daily.

5.11 Biological seed.

6. Glassware and Dilution Water Preparation

6.1 All dilution water and reagent storage bottles, BOD incubation bottles, and other glassware must be free of organic contaminants and toxic metals. Clean all glassware with hot soapy water, rinse with 3 N HCl, rinse three times with hot tap water and twice with distilled water. Any glassware with a film should not be used.

6.2 The distilled water should be cooled to 20°C, saturated with oxygen by bubbling air through the water and then stored at 20°C until use. Just prior to using the dilution water, add 1 ml each of the magnesium sulfate, calcium chloride, ferric chloride, and phosphate buffer solution solutions for each liter of water. The biological seed should be added (5 ml seed/l of dilution water) to the dilution water just before use.

7. Selection of Seed

7.1 All chlorinated domestic wastes and most industrial wastes require seeding because of low microbial populations. The standard seed material is primary treated sewage that has been stored at 20°C for 24 hours. However, it is important that, if possible, the seed to be used has been exposed to the waste that is being measured. Therefore, an effluent from a treatment process or a receiving water collected below the outfall will sometimes be used as seed material.

8. Interferences and Pretreatment of Samples

8.1 Blend samples containing non-homogeneous particulate matter with the Tekmar SDT Tissuemizer. Thirty seconds is usually adequate.

8.2 Neutralize samples with a pH outside of the range 5-10 using the 1 N acid or base. Most samples do not require neutralization because the buffering capacity of the dilution water and dilution of the samples.

8.3 Residual chlorine kills the seed organisms. All samples except those known not to contain residual chlorine should be checked as follows: Add 5 ml of 1 N H_2SO_4, 2 g KI crystals and 1 ml of starch solution to 100 ml of sample. Add the 0.028 N sodium sulfite solution in 0.1 ml increments until the purple color disappears. Each 0.1 ml increment corresponds to 1 mg/l Cl_2. Add a proportional volume of 0.028 N sodium sulfite to an aliquot of sample for testing. If there is any uncertainty, add an extra increment of sulfite. An excess of sulfite solution of 1 ml/l sample causes a BOD of less than 0.5 mg/l, which is insignificant.

8.4 Many organic compounds and trace metals are toxic to the seed organisms. Sometimes this interference can be eliminated by sample dilution. Higher BOD values from the more dilute aliquots is evidence of sample toxicity. These results should be carefully evaluated before being reported.

8.5 Samples containing more than 9 mg/l DO at 20°C may be encountered during winter months or in localities where algae are growing actively. To prevent loss of oxygen during incubation of these samples, reduce the DO to saturation by bringing the sample to about 20°C in a partly filled bottle and agitating it by vigorous shaking.

9. Calibration of Dissolved Oxygen Meter

9.1 Carefully fill 3 BOD bottles by use of a siphon with dilution water (containing nutrients but not seed) that has been saturated with air at 20°C. Using the table in the DO meter manual, find the DO concentration at the ambient atmospheric pressure and 20°C. Set the temperature dial on the meter if necessary to 20°C and adjust the calibration knob until the meter reads the value determined from the table. Save the other two bottles for checking the meter during the analysis

9.2 Drifting of the meter response or a very slow response to DO changes is usually caused by a coated or torn electrode membrane.

10. Sample Analysis Procedure

10.1 Since most samples require more than 7 mg/l of O_2 for stabilization, dilutions are required before incubation. Prepare a sufficient number of dilutions so that at least one aliquot depletes at least 2 mg/l and has a residual DO of at least 1 mg/l after incubation. Usually three and sometimes four dilutions are required. Dilutions up to 1% are made directly in the BOD bottles. A guide to sample size selection follows:

Measurable BOD Range	Sample Size, ml	Factor	% Dilution
4 - 12	150	2	50
8 - 24	75	4	25
12 - 36	50	6	16.67
20 - 60	30	10	10
40 - 120	15	20	5
60 - 180	10	30	3.33
120 - 360	5	60	1.67
200 - 600	3	100	1

For dilutions less than 1%, the sample is first diluted 1/10 or 1/100 with dilution water and then the dilutions are completed in the BOD bottles. The samples should be homogenized and shaken just before aliquots are taken. A graduate cylinder is used to measure volumes of 15 ml or larger. Large bore pipets are used for smaller volumes. One bottle per dilution is prepared. Exercise care in filling the bottles with dilution water so as not to have the water into the neck of the bottle more than 1/8".

10.2 Prepare two bottles with seeded dilution water. Depletion of these samples should be about 0.6 mg/l if domestic sewage is used for seed. Blank values over 1.0 mg/l indicates contaminated dilution water or incubation bottles.

10.3 Prepare one bottle with 5 ml of glucose-glutamic acid standard A and one bottle with 10 ml of standard B and fill with seeded dilution water. The results for standards A and B should be about 200 and 160 mg/l, respectively.

10.4 Measure the initial DO of all samples, being careful not to displace any of the dilution water. At the same time the DO is measured, the probe mixes the samples. Wash the probe with distilled water between each sample. After determining the DO it may be necessary to add a small amount of dilution water to prevent trapping bubbles in the bottle when stoppering. Place a water seal in the neck of the bottle and place a cap over the neck to maintain the water seal.

10.5 It is helpful to measure the DO of the samples after two days in order to judge the adequacy of the dilutions selected. Pour off the water seal before measuring the DO. Calibrate the DO meter according to the directions given in Section 9. Measure the DO of the most concentrated dilution of each sample. If there is less than 2 mg/l residual DO, increase the dilution factors on subsequent days and measure the DO in the next most dilute sample. If the DO on the second sample is less than 4 mg/l, re-aerate with an air stone attached to an air pump being careful not to displace any of the water. Record the initial residual and re-aerated DO values. Discard any sample with a residual DO below 1 mg/l. If there is less than a 2 mg/l depletion, increase the strength of the dilutions on subsequent days.

10.6 The final DO measurements are made within 4 hours of 5 days of when the samples were set up. Calibrate the DO meter by the method given in section 9. Any dilutions resulting in residual DO's that are 1 mg/l or greater and depletions that are 2 mg/l or greater are valid. Calculate the BOD values by the following formula:

$BOD_5 = F[(D_i - D_f) - f(B)]$

where D_i = initial DO of sample, mg/l

D_f = final DO of sample, mg/l

B = the mean depletion of the two seeded dilution water blanks, mg/l

f = decimal fraction of dilution water in sample bottle

F = whole number dilution factor of sample

For example, 30 ml of sample was used, the initial DO was 8.2 mg/l and the final DO was 1.7 mg/l. The initial DO of both of the seeded blanks was 8.1 mg/l and the final DO was 7.3 mg/l

$BOD_5 = 10[(8.2-1.7)-0.9(8.1-7.3)]$

$= 10[6.5-0.9(0.8)]$

$= 10[6.5-0.7]$

$= 10[5.8]$

$= 58$ mg/l

10.7 Report the average value of all of the valid dilutions to the nearest whole number with at most two significant figures. If the DO depletions increase with increasing dilution, toxicity is indicated and the results should be carefully evaluated before being reported.

10.8 The results of the A&B glucose-glutamic acid standards should be between 160-240 and 130-190 mg/l, respectively. High results indicate a very efficient seed or contaminated samples. Low results indicate a poor seed or blank values that were too high.

10.9 The mean of the seeded dilution water blank depletions should be below 1 mg/l, ideally 0.6 mg/l. High values indicate contaminated nutrients and minerals, dilution water or glassware. Correct any problems before proceeding.

10.10 Report the BOD values from different dilutions as duplicates on the AQC sheets.

10.11 Attach the incubator temperature recorder chart to the BOD Data/ Calculation Sheet (attached).

Prepared by M. Carter 6/9/78

BOD Data/Calculation Sheet, Rev. 6/9/78

Analyst _____ Study _____ Date/Time In _____
Date/Time Out _____

ample No.													
ample pH													
ample vol, ml													
nitial DO, mg/l													
-day DO, mg/l													
e-aerate DO, mg/l													
Final DO, mg/l													
ross O_2 dep., mg/l													
Blank Corr., mg/l													
et O_2 depl., mg/l													
Factor													
BOD_5, mg/l													
ean BOD_5, mg/l													
ample No.													
ample pH													
ample vol, ml													
nitial DO, mg/l													
-day DO, mg/l													
e-aerate DO, mg/l													
inal DO, mg/l													
ross O_2 depl., mg/l													
lank Corr., mg/l													
et O_2 depl., mg/l													
Factor													
BOD_5, mg/l													
ean BOD_5, mg/l													

DO Probe Calibration

	temp horr.	press.	DO, mg/l
initial			
2-day			
5-day			

ATTACHMENT II

TOTAL SUSPENDED SOLIDS

STORET NO. 00530

1. Scope and Application
   1.1 The method is applicable to drinking, surface and saline waters, and to domestic and industrial wastes.
   1.2 The detection limit of the method is 1 mg/l.

2. Summary of Method
   2.1 A homogenized sample is filtered through a pre-washed glass fiber filter. The residue retained on the filter is washed and then dried to constant weight at 105°C and weighed to the nearest 0.1 milligram. The TSS is calculated from the amount of residue per unit volume of sample.
   2.2 The filtrate from this method may be used to determine the total dissolved solids.

3. Sample Handling and Preservation
   3.1 Samples should be stored at 4°C and analyzed as soon as possible, but no later than 7 days after collection.

4. Apparatus
   4.1 Whatman GF/C glass fiber filter discs, 43 mn.
   4.2 Millipore membrane filtering apparatus with reservoir and a coarse fritted disc as a filter support.
   4.3 Aluminum drying pans, 50 mm and metal tray.
   4.4 Tekmar SDT Tissuemizer.
   4.5 Drying oven, 103°-105°C.
   4.6 Desiccator, with Drierite indicating desiccant.
   4.7 Analytical balance, 160 g capacity or larger, sensitive to 0.1 mg and one weight equivalent to the optical range of the balance.
   4.8 Graduate cylinder and wide bore pipets.

5. Balance Calibration
   5.1 Using a balance with an optical range of 1.0 g, place a 1.0 g (I5%) weight on the balance pan, set the weight control knob to 1.0 g, release the balance and set the zero point with the optical zero knob. With the balance released, slowly turn the weight control knob back to zero. The optical scale should come to rest exactly at 1.0 g. If the reading is more or less than 1.0 g, arrest the balance, remove the top housing cover and adjust the sensitivity weight. Repeat the calibration check.

6. Procedure
   6.1 Preparation of glass fiber filter disc: Place the glass fiber filter on the membrane filter apparatus with wrinkled surface up. While vacuum is applied, wash the disc with 100 ml of distilled water. Remove all traces of water by continuing to apply vacuum after water has passed through. Remove filter from membrane filter apparatus, place in aluminum pan, and dry in an oven at 103-105°C for one hour. Remove to desiccator and store until needed. Weigh immediately before use. After weighing, handle the filter with forceps only.

6.2 Homogenize all non-uniform samples with blender and shake the bottles before withdrawing an aliquot to assure taking a representative sample.

6.3 Choose a maximum sample volume that will filter in 5 minutes or less. Measure volumes smaller than 15 ml with wide bore pipets and larger volumes with graduate cylinders. Discard any sample which does not filter in 5 minutes and filter a smaller sample volume.

6.4 Wash the graduated cylinder or pipet and with the suction on, wash the filter funnel wall, filter and residue with two twenty-five ml portions of distilled water allowing complete drainage between washings. Remove all traces of water by continuing to apply vacuum after water has passed through.

6.5 Carefully remove the filter from the filter support. Place in an aluminum pan and dry at least one hour at 103-105°C. Cool and weigh immediately or place in a desiccator for later weighing. Re-dry and re-weigh 10% or at least one filter per set of samples. If the incremental weight loss is less than 0.5 mg, calculate the results based on the original weights. If the weight loss exceeds 0.5 mg, re-dry and re-weigh all of the filters and re-check 10% of the filters.

6.6 Analyze two blanks per set of samples by filtering 100 ml of distilled water through two prepared filters. The amount of additional weight loss after the filters have been prepared is nearly independent of the volume of water filtered. Therefore, add the mean blank weight loss to the residue weight for each sample.

6.7 Analyze 10% or at least one sample per set in duplicate.

6.8 Analyze a standard sample with each sample set.

6.9 Calculate the results as follows:

$$TSS = \frac{(W_G - W_T) + B}{V_S}$$

W_G = Gross weight of filter and residue, mg

W_T = Tare weight of filter, mg

B = The mean of the two blank results, mg

Where $B = \frac{B_1 + B_2}{2}$

$B_1 = B_T - B_G$

B_T = Tare weight of filter, mg

B_G = Gross weight of filtering

V_S = Volume of sample filtered, l

TS[redacted]ATA/[redacted]CUL[redacted]N S[redacted]T 6/[redacted]B

Analyst _____ Study _____ Date/Time Filters in Oven _____
Date/Time Out _____

Sample No.										
Sample Vol., l										
Re-check Wt. mg										
Gross Wt., mg										
Tare Wt., mg										
Residue Wt., mg										
Blank Corr., mg										
Corr. Res. Wt., mg										
TSS, mg/l										
Sample No.										
Sample Vol., l										
Re-check Wt., mg										
Gross Wt., mg										
Tare Wt., mg										
Residue Wt., mg										
Blank Corr., mg										
Corr. Res. Wt., mg										
TSS, mg/l										
Sample No.										
Sample Vol., l										
Re-check Wt., mg										
Gross Wt., mg										
Tare Wt., mg										
Residue Wt., mg										
Blank Corr., mg										
Corr. Res. Wt., mg										
TSS, mg/l										

Balance Calibration

	Reading on 100 mg weight, mg
Tare	
Gross	
Re-check	

ATTACHMENT III

Analysis of Organic Pollutants in Water by Direct Aqueous Injection, Gas Chromatography-Mass Spectrometry

NEIC - September 1978

1.0 Introduction

1.1 Many volatile organic compounds are soluble in water at concentrations exceeding 1 mg/l. However, they are not suitable for Volatile Organics Analysis (V.O.A.) due to their low purgeability. This method is suitable for GC/MS identification, confirmation, and quantitation of the previously mentioned types of compounds.

2.0 Summary of Method

2.1 A sample is injected into the inlet system of a gas chromatograph. After vaporization, the aqueous sample is carried through a column by an inert carrier gas. The sample components are partitioned between the carrier gas and a stationary liquid phase on an inert solid support. The column effluent is introduced into a quadrupole mass spectrometer by means of a glass jet separator. From the interface, the sample is passed into an electron impact ionization source. The various ion fragments are filtered by a quadrupole mass filter and detected by a continuous dynode electron multiplier. The signal is then fed to a computer controlled data system for processing. Compounds are matched with standard spectra stored in a library and identified based upon their spectral similarity and relative retention times. Concentrations are calculated for each identified compound based upon its relative response to an internal standard.

3.0 Interferences

3.1 Particulate matter - Particulate or suspended matter should be removed to prevent both plugging of syringes and formation of condensation nuclei. Allowing particulates to settle before analysis is acceptable.

3.2 Stability - Aqueous solutions of D-chloroform ($CDCl_3$) are unstable. The $CDCl_3$ can exchange to $CHCl_3$.

Stock standards of deuterochloroform (7500 ng/ul) that are prepared 24 hours prior to dosing and analysis, display large losses in response.

Even stock standards prepared 8 hours prior to dosing and analysis, exhibit some loss of response. Stock standards of D-chloroform are prepared in vials that have approximately two ml. head space. Volatility losses occur in this head space. No losses are observable if the stock standard solution is refrigerated and used within four hours of preparation.

3.3 Identical Retention Times - It is possible with any given column and operating conditions, to have two compounds that elute at identical retention times. It is especially important to choose an internal standard that does not coelute with another compound of interest. This problem is minimized by using GC/MS.

4.0 Apparatus

4.1 Finnigan 3200 Gas Chromatograph/Mass Spectrometer System with a Finnigan INCOS data system and Revision 3.1 software (1).

5.0 Reagents and Materials

5.1 Purity of Reagents - All chemicals used for standards and internal standards shall be of the highest purity available.

5.2 Purity of Water - All water shall be of sufficient purity such that no background is observed above the detection limit of the compounds of interest. Filtration through activated carbon will eliminate any interferences.

5.3 Carrier Gas - Only high purity helium shall be used.

5.4 Column

5.4.1 Column Tubing - Stainless steel, oil free. Dimensions 1/8" OD x 20'.

5.4.2 Solid Support - Chromosorb W acid washed 80/100 mesh.

5.4.3 Liquid Phase - Carbowax 20m - 5% loading.

5.5 Internal Standard - Dilute 50 ul of deuterochloroform to 10 ml with water. Shake well to assure all D-chloroform is in solution. The concentration of this solution is 7500 ng/ul.

5.5.1 Prepare this solution fresh every four hours and keep refrigerated.

5.6 Standards

5.6.1 Concentrated Standards - Prepare stock standards of each compound of interest by weighing out 50 mg of pure compound and diluting this with water to a volume of 50 ml. Stability of stock solutions is enhanced by keeping the solutions refrigerated. Stock solutions should be prepared fresh every two weeks.

5.6.2 Analytical Standards for GC/MS - Dilute the concentrated standards by adding 0.5 ml of each concentrate to a 12 ml vial and bringing the volume to 10 ml. This working standard should be prepared each day. Each ul of working standard is equal to 50 ng (50 ng/ul).

5.7 Mass Spectrometer Performance Standard - Prepare a 150 ng/ul aqueous solution of Pentafluorobromobenzene and refrigerate until ready for use. This solution is stable for one month.

6.0 Samples and Sampling Procedure

6.1 Sample Collection - Samples should be collected so that no air remains in the bottle as a head space once the vial cap is tightened.

6.2 Sample Containers - 1 oz. glass bottles equipped with Teflon lined silicone septa and screw caps (Pierce #13074 and #12722 or equivalent). Before sampling, wash used bottles with soap (Alconox or equivalent) and tap water, rinse with tap water. New bottles require only washing with tap water. Bake bottles at 200°C and septa at 80°C for 30 minutes. Allow to cool in a desicator with charcoal adsorbant to maintain an organics free atmosphere. Then cap the bottles and hold for sampling.

6.3 Sample Size for Analyses - The sample size must be small to prevent overloading of the column. For aqueous analysis, a sample size of 5 ul is optimum.

6.4 Sample Storage - Storage time of samples should be kept to a minimum. If storage cannot be avoided, the bacterial action, as well as volatility losses, should be minimized by refrigeration (2).

7.0 Procedure

7.1 Mass Spectrometer Calibration

7.1.1 Adjust and calibrate the mass spectrometer according to the manufacturers specifications.

7.1.2 Analyze a sample of pentafluorobromobenzene (PFBB).

7.1.3 Determine if the PFBB spectrum meets the performance criteria (3) (Attachment 1). Proceed to analyses if it does or retune the instrument to meet the performance criteria.

7.1.4 Analyze a standard mix of the compounds of interest and determine if the response is within an acceptable range of the previously established response factors. If not, determine the cause of the problem, make the necessary corrections and reanalyze the standard.

7.2 Sample Analysis

7.2.1 Equilibrate the sample bottles to ambient temperature and pipette one ml of sample into a 12 ml vial. Composite samples may be prepared by pipetting one ml volumes of each sample into a 12 ml vial. Dose the sample (composite) with 10 ul of internal standard solution for each one ml of sample to yield an internal standard concentration of 75 ng/ul.

7.2.3 Equilibrate the GC oven temperature to 70°C.

7.2.4 Inject 5 ul of the dosed sample, turn the vacuum diverter off and immediately start collecting M.S. data using the following conditions:

Mass Range 33 - 130 AMU
Scan Time - 3 seconds
After four minutes start the G.C. oven program (6°/min) oven max = 180°C

7.2.5 Collect data until the last components have eluted from the G.C. column. Typically this would be 320 scans or about 16 minutes.

7.3 Data Evaluation

7.3.1 After each analysis, collected data is analyzed by the procedure - Computer Assisted Evaluation of Direct Aqueous Injection GC/MS Data (4).

References

(1) Finnigan INCOS Data System Operators Manual, Revision 3; Finnigan 3200 GC/MS Systems Manual, Finnigan Corporation, Sunnyvale, California

(2) "Standard Recommended Practice for Measuring Volatile Organic Matter in Water by Aqueous-Injection Gas Chromatography," ASTM D-2908-74, p 480-487

(3) Memo of J. Eichelberger and W. Budde, March 10, 1978, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, Subject - Perfluorobromobenzene Reference Compound for Use with Typical Purge and Trap Columns that Do Not Transmit DFTPP Readily.

(4) Computer Assisted Evaluation of Direct Aqueous Injection GC/MS Data - Procedure Developed by the Chemistry Branch of the EPA, National Enforcement Investigations Center, Denver, Colorado, September 1978

ATTACHMENT IV

Computer Assisted Evaluation of
Direct Aqueous Injection GC/MS Data

NEIC - September 1978

1.0 Introduction

1.1 This procedure is a slightly modified version of the priority pollutant data evaluation procedure (1). Minor modifications were made to enhance the handling of direct aqueous injection (DAI) analyses data for the Kanawha River Valley project (August 1978).

2.0 Summary of Method

2.1 GC/MS data files are processed by location of an internal standard that is used for response reference. Compounds of interest in a user library are reverse searched using an absolute retention time window. If a compound is located and passes the match criteria, it is quantitated and the spectrum printed. Printed results are manually audited and the data verified or rejected.

3.0 Summary of Modifications

3.1 The compound detection routine (Detect) was changed to use absolute retention times for location of the retention time window. Only masses 41 through 125 were used in locating compounds due to the Argon background (m/e 40) in the system.

3.2 The required spectrum match parameter limit (fit) in the compound identification routine (Detec 2) was set to 450. This lower limit was necessary due to the poor character of the spectra of the DAI compounds. Poor character here means that the spectra contain few ions and their response (sensitivity) is poor.

3.3 The names of procedures used in both the DAI data evaluation and the priority pollutant evaluation were changed to allow independent operation of the two procedures.

4.0 Interferences

4.1 In some cases, a spectrum may match the library reference sufficiently to be passed. During quantitation, however, the ion of interest may be too weak to locate and no entry will be made in the quantitation list. In such a case, no entry at all (e.g. no "not found" entry) will appear in the quantitation report. The name and match results will, however, appear in the qualitative data report.

4.2 Occasionally, multiple peaks will be detected during quantitation due to background interferences and multiple entries will be made in the quantitation list. Generally, the entry having the same label as the correct spectrum is used for quantitation and the others are disregarded. In some instances, however, the correct selection is not obvious and manual evaluation of the quantitation results must be done.

5.0 Apparatus

5.1 Finnigan INCOS data system software, Revision 3.1 or later. To initially set up this procedure, the user must understand and be proficient in the use of MSDA (2).

6.0 Procedure

6.1 Procedure Set Up

6.1.1 Create the procedures from the trace of EVDAI in Appendix I.

6.2 Library Set Up

6.2.1 Build a user library containing each compound of interest. Appendix II is a library list of the DI library. The first entry must always be the internal standard and each entry must include the quantitation parameters and retention times.

6.2.2 Execute EVDAI, edit the quantitation list for accuracy and update the library parameters using commands in "QUAN".

6.2.3 Using the "LIBR" program, generate hard copies of library spectra for reference. Using the library list editor, "EDLL", generate summaries of the entries and quantitation parameters as in Appendix II.

6.3 Routine Use

6.3.1 Analyze samples, standards and quality control samples using the same instrument conditions used to set up the libraries.

6.3.2 Using the namelist editor, create a namelist containing the names of the data files to be processed.

6.3.3 Execute the procedure as follows:

EVDAI library, namelist, yes (no)

Where: library is the appropriate user library name.

namelist is the list containing the files to be processed.

yes (no) selects printout of the spectra at a peak that was identified by the procedure.

6.3.4 Appendix III is an example of PPEVAL output. The "No" option was selected.

7.0 Quality Control

7.1 Each identification can be manually audited if the "yes" option was selected. Inaccurate qualitative results may then be checked and manually corrected.

7.2 Quantitation data accuracy is monitored by use of standard quality control techniques such as daily standardization, replicate analysis and spikes (3). Daily calibration of the method can be accommodated by analyzing the standard data first, updating the relative response factors, obtaining hard copy of the new factors (library list editor) and then analyzing sample data.

8.0 Precision and Accuracy

8.1 The overall precision and accuracy is limited to the quality of the raw data being processed.

9.0 References

(1) "Computer Assisted Evaluation of Organic Priority Pollutant GC/MS Data", US EPA, National Enforcement Investigations Center, September 1978.

(2) "INCOS Data System - MSDS Operators Manual - Revision 3", Finnigan Instruments, March 1978.

(3) "Quality Assurance Program for the Analyses of Chemical Constituents in Environmental Samples", US EPA, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio, March 1978.

APPENDIX Ia.

```
TRACE OF PROCEDURE EVDAI
   * ERASE
   * ;[********** PRIORITY POLLUTANT EVALUATION PROCEDURE **********]
   * ;[THIS PROCEDURE MAY BE USED TO EVALUATE GC/MS DATA             ]
   * ;[FOR PRIORITY POLLUTANT (EPA SECTION 307(A)) COMPOUNDS         ]
   * ;[THE PROCEDURE UTILIZES INTERNAL STANDARDS AND RELATIVE        ]
   * ;[RESPONSE FACTORS FOR QUANTITATION.  THE MSDS OPTION           ]
   * ;[SEARCH IS USED TO LOCATE AND IDENTIFY PEAKS.  THE EPA         ]
   * ;[IDENTIFICATION CRITERIA, E.G., THREE IONS PER COMPOUND        ]
   * ;[.IS USED TO LOCATE THE COMPOUND OF INTEREST.  MORE IONS       ]
   * ;[HOWEVER MAY BE USED AS THE FIT OF THE SEARCH ROUTINE WILL     ]
   * ;[YIELD MORE SPECIFICITY FOR THE COMPOUND.  THE FULL            ]
   * ;[SPECTRUM IS OUTPUT IN ORDER TO PROVIDE CONFIRMATION OF        ]
   * ;[THE PRESENCE OF THE COMPOUNDS.                                ]
   * ;[**************************************************************]
   * ;[TO USE PPEVAL, BUILD A LIBRARY CONTAINING THE SPECTRA OF      ]
   * ;[THE COMPOUNDS OF INTEREST.  INCLUDE THE QUANTITATIVE DATA     ]
   * ;[THAT IS NECESSARY AS DESCRIBED IN THE MSDS MANUALS.           ]
   * ;[CREATE A NAMELIST WITH THE NAMES OF THE FILES TO BE           ]
   * ;[PROCESSED.  EXECUTE THE PROCEDURE AS FOLLOWS:                 ]
   * ;[    PPEVAL LIBRARYNAME, NAMELIST  E.G. PPEVAL V0.SAMPLE      ]
   * ;[  REVISED 30AUG78 D.J.LOGSDON II  EPA-NEIC 303-234-4661      ]
   * ;SETS PPSCAN;EDLL YES(-;S;W;E);EDLL NO(-;W;E)
   * ;SETN S2;SET4 S1;PPEV1;FEED;BEEP;BEEP;BEEP
   *
   ERASE
   SETS PPSCAN
   EDLL YES (-;S;W;E)
   EDLL NO (-;W;E)
   SETN S2
   SET4 S1
   PPEV1
      * ERASE
      * ;[PART OF PROCEDURE PPEVAL                                   ]
      * ;[GET THE NEXT NAMELIST ENTRY AND CONTINUE PROCESSING        ]
      * ;[AT PPEV2                                                   ]
      * ;GETN;PPEV2;LOOP
      *
      ERASE
      GETN
      PPEV2
         * ERASE
         * ;[PART OF PPEVAL.  THIS PROCEDURE SETS THE LIBRARY ENTRY          ]
         * ;[POINTER TO THE FIRST ENTRY, WHICH MUST ALWAYS BE THE INTERNAL    ]
         * ;[STANDARD.  LOCIS IS THEN CALLED AND THE INTERNAL FOUND           ]
         * ;[THE SPECTRUM NUMBER OF THE INTERNAL STANDARD IS                  ]
         * ;[STORED IN !10 FOR FUTURE REFERENCE.  THE LIBRARY POINTER         ]
         * ;[IS THEN RESET TO THE BEGINNING, THE QUANTITATION LIST SET TO     ]
         * ;[THE FILE NAME AND EMPTIED OUT.  DETECT IS CALLED TO LOCATE EACH  ]
         * ;[COMPOUND (IF PRESENT).  QUAN IS THEN CALLED TO CALCULATE         ]
         * ;[THE RESULTS AND THE PROCEDURE RETURNS TO PPEV1 TO GET THE        ]
         * ;[NEXT FILE TO PROCESS.                                           ]
         * ;FILE(K PRIN.99/N;E)
         * ;EDLL PPLIST(-;W;E)
         * ;SETJ #1;CHRO(I;H1,900,300;E);SET4 #1;LOCIS;SET10 !14;SET4 #0
         * ;SETO S1;EDOL(-;W;E);EDSL(-;W;E);SETL S3;DETECT;QUAN(I;H;E)
         * ;EDLL PPLIST(0!1;E)
         * ;PRINTQPP)
         * ;FILE(C PRIN.99/N,M::E)
         * ;FEED
         * ;BEEP
         *
         ERASE
```

APPENDIX Ib.

```
   * ;[PART OF PPEVAL                                                    ]
   * ;[ROUTINE TO FIND AN INTERNAL STANDARD IN A SAMPLE    ]
   * ;[USE A REVERSE SEARCH TO LOCATE THE INTERNAL STANDARD]
   * ;SET14 #0
   * ;SEAR/V(I;S;V2500000;N2,10,400;&;D-60,60;E)
   * ;LOCIS1
   *
   ERASE
   SET14
   SEAR  (I;S;V2500000;N2,10,400;&;D-60,60;E)/V
   LOCIS1
      * IF LOCIS1 ,!14
      * ;[PART OF PPEVAL            ]
      * ;[NO INTERNAL STANDARD FOUND]
      * ;PRIN(@IS)
      * ;RETU PPEV2
      *
      IF LOCIS1,!14
      PRIN  (@IS)
      RETU PPEV2
SET10 !14
SET4
SET0 $1
EDDL  (-;W;E)
EDSL  (-;W;E)
SETL $3
DETECT
   * ;[PART OF PPEVAL                                                   ]
   * ;[THIS ROUTINE LOCATES COMPOUNDS IN THE                           ]
   * ;[SAMPLE FILE BY COMPARING THE SPECTRA IN THE LIBRARY             ]
   * ;[WITH THE SAMPLE.  RELATIVE RETENTION TIMES ARE USED             ]
   * ;[AND REFERENCED TO THE INTERNAL STANDARD FOUND EARLIER.]
   * ;[THE LIBRARY POINTER IS BUMPED AND TESTED TO                     ]
   * ;[SEE IF THE LAST LIBRARY ENTRY HAS BEEN PROCESSED.               ]
   * ;[THEN THE CURRENT SCAN NUMBER IS SET TO THE INTERNAL             ]
   * ;[STANDARD LOCATION BY RECALLING THE CONTENTS OF !10.             ]
   * ;[STORE THE SCAN NUMBER OF                                        ]
   * ;[THE BEST MATCH IN VARIABLE 14 AND ALLOW INTEGRATION             ]
   * ;[AT THAT SPECTRUM NUMBER ONLY                                    ]
   * ;[IF THE COMPOUND IS NOT FOUND, PLACE A NOT FOUND                 ]
   * ;[ENTRY INTO THE QUANTITATION LIST FOR LATER REFERENCE            ]
   * ;SET4 !4,,#1
   * ;IF !24#1,!4
   * ;SET14 #0
   * ;SET1 !10
   * ;EDLL PPLIST(S;W;E)
   * ;SEAR/V(I;S;&;V2500000;M41,125;N1,10,10;D-20,20;E)
   * ;PRIN/KX(14,2;!14,6;!15,6;!16,7;C;E)
   * ;DETEC1
   * ;LOOP
   *
   SET4 !4,,#1
   IF #1!24,!4
   SET14
   SET1 !10
   EDLL PPLIST (S;W;E)
   SEAR  (I;S;&;V2500000;M41,125;N1,10,10;D-20,20;E)/V
   PRIN  (!4,2;!14,6;!15,6;!16,7;C;E)/KX
   DETEC1
      * [PART OF PPEVAL]
      * ;[IF THE FIT IS LESS THAN OR EQUAL TO 750    ]
                                                        TUE]
```

APPENDIX Ic.

```
                    * ;[DATA IN THE QUANLIST ASSIGNED EARLIER.  ]
                    * ;[ALSO CHECK AND PASS ONLY PEAKS WITH     ]
                    * ;[A FIT OF 750 OR GREATER                 ]
                    * ;IF DETEC2 !16,DETEC2 @450
                    * ;SET1 !14
                    * ;CHRO(I;R;S;@;N1,3;A>5,3;G-4,4;D-5,5;E)
                    * ;DETEC3
                    * ;RETU DETEC1
                    *
                    IF DETEC2!16,DETEC2@450
                    SET1 !14
                    CHRO  (I;R;S;@;N1,3;A>5,3;G-4,4;D-5,5;E)
                    DETEC3
                       * IF !26 DETEC3,DETEC3
                       * ;SPEC(*;N;H;E)
                       *
                       IF DETEC3!26.DETEC3
                       SPEC  (*;N;H;E)
                    RETU DETEC1
                 EDOL  (-;N;@;A;E)
              LOOP
           QUAN  (I;H;E)
           EDLL PPLIST (B!1;E)
           PRIN  (OPP)
           FILE  (C PRIN.99/N,M:;E)
           FEED
           BEEP
        LOOP
FEED
BEEP
BEEP
BEEP
```

APPENDIX IIa.

NAM NUM: NAME WT FORMULA REL.RET.TIME/CAS#	MASS	RET TIME AMT.	BASE REF.PEAK	AREA RESP.FILE	U.P.#1 RESP.FACTOR	U.P.#2
DI 1: D-CHLOROFORM						
119		7:27	84	91648.	0.000	0.000
1.000	84.000	75.00 DI	1	:S	1.000	
DI 2: ETHYL ETHER						
74 C4.H10.O		2:27	59	37312.	0.000	0.000
0.358	45.000	50.00 DI	1	:S	0.333	
DI 3: ACETONE						
58 C3.H6.O		3:27	43	175872.	0.000	0.000
0.504	43.000	50.00 DI	1	:S	1.201	
DI 4: METHYL ETHYL KETONE						
72 C4.H8.O		4:27	43	202240.	0.000	0.000
0.659	43.000	50.00 DI	1	:S	1.682	
DI 5: ACRYLONITRILE						
53 C3.H3.N		6:57	53	26209.	0.000	0.000
0.933	53.000	50.00 DI	1	:S	0.446	
DI 6: STYRENE						
104 C8.H8		13:33	104	63809.	0.000	0.000
1.978	104.000	50.00 DI	1	:S	0.951	
DI 7: 1,1 DIMETHOXYETHANE						
90 C4.H10.O2		3:30	59	134144.	0.000	0.000
0.470	75.000	50.00 DI	1	:S	0.331	
DI 8: ISOPROPANOL						
60 C3.H8.O		5:15	45	84736.	0.000	0.000
0.705	45.000	50.00 DI	1	:S	1.034	
DI 9: DIETHYL KETONE						
86 C5.H10.O		6:36	57	84992.	0.000	0.000
0.886	86.000	50.00 DI	1	:S	0.313	
DI 10: ISOBUTRONITRILE						
69 C4.H7.N		7:19	42	59904.	0.000	0.000
0.980	42.000	50.00 DI	1	:S	0.928	
DI 11: N-BUTANOL						
74 C4.H10.O		11:06	56	14080.	0.000	0.000
1.490	56.000	50.00 DI	1	:S	0.162	
DI 12: PROPANE,2,2'-OXYBIS						
102 C6.H14.O		2:42	45	134912.	0.000	0.000
0.362	45.000	50.00 DI	1	:S	0.857	
DI 13: 1,3-DIOXOLANE,2-METHYL						
[illegible]		[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
0.805	73.000	50.00 DI	1	:S	0.774	
DI [illegible]						
92 C4.H9.CL		4:03	56	15230.	0.000	0.000
0.544	56.000	50.00 DI	1	:S	0.211	

APPENDIX IIb.

NAM NUM: NAME WT FORMULA REL.RET.TIME/CAS#	MASS	RET TIME AMT.	BASE REF.PEAK	AREA RESP.FILE	U.P.#1 RESP.FACTOR	U.P.#2
DC 1: DCHLOROFORM						
119		7:15	84	163348.	0.000	0.000
1.000	84.000 75.00	DC	1	:S	1.000	
DC 2: ETHANOL						
46 C2.H6.O		5:18	45	106624.	0.000	0.000
0.731	45.000200.00	DC	1	:S	0.235	
DC 3: MESITYL OXIDE						
98 C6.H10.O		10:35	83	93568.	0.000	0.000
1.462	83.000220.00	DC	1	:S	0.219	
DC 4: ETHANOL,2-METHOXY-,ACETATE						
118 C5.H10.O3		13:57	43	565600.	0.000	0.000
1.924	58.000200.00	DC	1	:S	0.731	

APPENDIX IIIa.

QUANTITATION REPORT FILE: D90579E

DATA: D90578E.TI
09/05/78 9:28:00
SAMPLE: MIX D 50NG/UL +IS
CONDS.: 70-180
FORMULA: @6/MIN INSTRUMENT: 3200EI WEIGHT: 0.000
SUBMITTED BY: JJS ANALYST: JJS ACCT. NO.: J240

AMOUNT=AREA * REF.AMNT/(REF.AREA* RESP.FACT)

NO	NAME
1	D-CHLOROFORM
2	ACRYLONITRILE
3	1,1 DIMETHOXYETHANE
4	ISOPROPANOL
5	DIETHYL KETONE
6	ISOBUTRONITRILE
7	N-BUTANOL
8	PROPANE,2,2'-OXYBIS
9	1,3-DIOXOLANE,2-METHYL
10	BUTANE,1-CHLORO

NO	M/E	SCAN	TIME	REF	RRT	METH	AREA	AMOUNT	%TOT
1	84	147	7:21	1	1.000	A BB	652404.	75.000 NG/UL	14.29
2	53	137	6:51	1	0.932	A BB	222688.	50.000 NG/UL	9.52
3	75	69	3:27	1	0.459	A BB	209334.	50.000 NG/UL	9.52
4	45	101	5:03	1	0.687	A BB	562915.	50.000 NG/UL	9.52
5	86	130	6:30	1	0.884	A BB	145856.	50.000 NG/UL	9.52
6	42	144	7:12	1	0.980	A BB	369855.	50.000 NG/UL	9.52
7	56	222	11:06	1	1.510	A BB	72404.	50.000 NG/UL	9.52
8	45	53	2:39	1	0.361	A BB	512704.	50.000 NG/UL	9.52
9	73	118	5:54	1	0.803	A BB	409852.	50.000 NG/UL	9.52
10	56	79	3:57	1	0.537	A BB	122668.	50.000 NG/UL	9.52

APPENDIX IIIb.

NAM	NUM:	WT	FORMULA	NAME
DI	1:	119		D-CHLOROFORM
DI	2:	74	C4.H10.O	ETHYL ETHER
DI	3:	58	C3.H6.O	ACETONE
DI	4:	72	C4.H8.O	METHYL ETHYL KETONE
DI	5:	53	C3.H3.N	ACRYLONITRILE
DI	6:	104	C8.H8	STYRENE
DI	7:	90	C4.H10.O2	1,1 DIMETHOXYETHANE
DI	8:	60	C3.H8.O	ISOPROPANOL
DI	9:	86	C5.H10.O	DIETHYL KETONE
DI	10:	69	C4.H7.N	ISOBUTRONITRILE
DI	11:	74	C4.H10.O	N-BUTANOL
DI	12:	102	C6.H14.O	PROPANE,2,2'-OXYBIS
DI	13:	88	C4.H8.O2	1,3-DIOXOLANE,2-METHYL
DI	14:	92	C4.H9.CL	BUTANE,1-CHLORO

IDENTIFICATION REPORT FILE: D:E90579E.TI

NO	SCAN	PURITY	FIT
1	148	378	996
2	49	351	728
3	73	344	661
4	97	410	740
5	0	0	0
6	277	268	929
7	73	73	207
8	97	123	260
9	148	43	323
10	137	37	133
11	217	110	533
12	49	185	459
13	106	42	360
14	97	164	329

ATTACHMENT V

Methodology: Carbaryl Analysis

A liter of the sample was extracted serially with three 50 ml portions of methylene chloride. The extracts were combined and passed through Na_2SO_4 into a 250 ml round bottom flask. 50 ml of ethyl acetate was added to the flask and the solvents were concentrated to 10 ml in a rotary evaporator at 45°C. The extract was passed through a clean-up column of 3 cm Florisil topped with 1 cm of Na_2SO_4. The Carbaryl was eluted with 20 ml of ethyl acetate. The 30 ml of ethyl acetate was concentrated to 10 ml on a hot plate under a gentle stream of carbon filtered air.

The extract was analyzed on a Waters 204 Liquid Chromatograph with a M Bondapak C_{18} column. A methanol - 1% acetic acid gradient was used over 25 minutes at a flow rate of 2.0 ml/min. The gradient was run from 0 to 80% methanol. The dual channel UV detector was operated at wave lengths of 254 nm and 280 nm.

Quality Control: A blank and a spike were analyzed along with the samples. The blank did not contain any interferences at the retention time of Carbaryl The spike was at a concentration of 250 ug/l of Carbaryl and the recovery was 117%.

The presence of Carbaryl in the samples was established by the coincidence of retention time and confirmed by the ratio of the 254 to 280 response.

ATTACHMENT VI

Neutral Extraction Technique for Organics Analysis
September 1978

1.0 Scope and Application

1.1 This procedure is applicable for analysis of water and wastewater samples for a broad spectrum of organic pollutants.

2.0 Summary of Method

2.1 Water and wastewater samples are extracted with CH_2Cl_2 (dichloromethane) at a neutral pH. The extract is dried and concentrated with the addition of acetone and iso-octane to exchange solvents. The resultant extract concentrate is subjected to GC and GC/MS analysis to identify and quantitate the organic pollutants present.

3.0 Sample Handling and Preservation

3.1 Prior to extraction, samples are refrigerated and extracted as soon as possible, generally within 48 hours. Samples may be held 5 days or more if necessary.

4.0 Definitions and Comments

5.0 Interferences

5.1 Solvents, glassware and reagents could be sources of contamination. Therefore, at least one "Reagent Blank" must be prepared contacting the solvent with all potential sources of contamination. This blank should then be processed through the same analytical scheme as the associated samples.

5.2 Typical interferences from reagents are: 4-methyl-4-hydroxy-2-pentanone (diacetone alcohol) from acetone, phthalate esters from Na_2SO_4, cyclohexene from dicholormethane.

6.0 Apparatus

6.1 Separatory funnels: 2l and 4l glass with glass or teflon stoppers and stopcocks. No stopcock grease used.

6.2 Drying column: All glass 3 cm x 50 cm with attached 250 ml reservoir.

6.3 Concentrator: 250 or 500 ml Kuderna-Danish evaporative concentrator equipped with a 5 or 10 ml receiver ampule and a 3 ball Snyder column.

7.0 Reagents

7.1 Extraction solvent: Pesticide analysis grade CH_2Cl_2 (dichloromethane) (Burdick and Jackson or equivalent)

7.2 Exchange solvents

7.2.1 Exchange solvent: Pesticide analysis grade acetone (Burdick and Jackson or equivalent)

7.2.2 Exchange solvent: Iso-octane suitable for pesticide analysis (Burdick and Jackson or equivalent)

7.3 Drying agent: Analytical reagent grade granular anhydrous Na_2SO_4 (sodium sulfate). Washed with CH_2Cl_2 prior to use.

7.4 Glass wool that has been extracted with CH_2Cl_2 prior to use.

7.5 6N NaOH for pH adjustment.

7.6 6N HCl for pH adjustment.

7.7 pH paper for pH measurement.

8.0 Procedure

8.1 If low concentrations of pollutants are expected, measure 3 l of sample for extraction. Otherwise, one l is sufficient.

8.2 Measure and record the initial pH. Adjust the pH to 6-8 if necessary, and record the adjusted pH.

8.3 Extract the sample with 3 successive extractions of 100, 50 and 50 ml of CH_2Cl_2 for 1 liter samples and 200, 100, 100 ml of CH_2Cl_2 for 3 liter samples.

If emulsions form, use a wire or stirring rod to break it, pass the emulsion through glass wool or centrifuge if necessary. Combine the extracts and measure the volume recovered. 85 percent constitutes an acceptable recovery.

8.4 Place a glass wool plug in a drying column and add ca 10 cm of Na_2SO_4. Wash the Na_2SO_4 with at least 50 ml of CH_2Cl_2. Pour the combined extract through the column. Follow with 100 ml of acetone. Collect the CH_2 and acetone and transfer to a KD assembly. Add 2 ml of iso-octane for 1 liter extracts and 5 ml iso-octane for 3 liter extracts.

8.5 Concentrate on a hot water bath at 80-90°C until the extract stops boiling. Quantitatively transfer the receiving tube contents to a graduated centrifuge tube. Adjust the volume to 2 or 5 ml by either adding more iso-octane or evaporating the excess iso-octane under a gentle stream of carbon filtered air. Transfer to a 12 ml vial and cap with a teflon lined cap. (Note: The final extract volume should depend on the sample. Extracts containing high concentrations of pollutants may not require concentrations to 5 ml while cleaner samples may require a final volume of 2 ml).

9.0 Quality Control

9.1 A representative group of the organic pollutants of interest should be spiked into water and carried through the extraction procedure, recoveries calculated and compared to literature values (if available).

10.0 Calculations

10.1 Solvent Recovery:
% recovery = Volume recovered (ml)*100/volume added (ml)

10.2 Pollutant Recovery:

$$\% \text{ recovery} = \frac{(\text{Concentration measured} - \text{initial concentration}) * 100}{\text{Concentration added}}$$

11.0 Precision and Accuracy

11.1 Precision and accuracy vary with the pollutants being measured. Recoveries range from 48 - 119 percent and precision values range from 1 to 9 percent relative standard deviation (% RSD). Typical values are ±5 % RSD.

12.0 References

(1) "An EPA GC/MS Procedural Manual-Review Copy", Environmental Monitoring and Support Laboratory, Cincinnati, Ohio.

ATTACHMENT VII

Summary of Recovery Data for Neutrals Extractable Organics in Kanawha River Project

Background

A number of organic compounds were identified in the Kanawha River Project reconnaissance samples. Some of these compounds were available and synthetic sample recoveries were measured to help validate the extraction methods used. Even though few of the compounds used in this evaluation were found in subsequent survey samples, the diversity of the compounds used illustrate the method's capability to recover a broad spectrum of pollutants.

Experimental

A standard mix was prepared containing 50 ng/ul of each compound in acetone. One and 3 l tap water samples were spiked with the standard mix resulting in concentrations of 2500 and 10 ug/l respectively. The samples were then extracted with CH_2Cl_2 and concentrated with the addition of iso-octane as an exchange solvent in Kuderna-Danish evaporative concentrators. The final volumes were 5 and 1 ml for the 1 and 3 l samples respectively. The extracts were then analyzed by gas chromatography with a flame ionization detector using a 6 ft x 2 mm glass column packed with 60/80 mesh GC-Q coated with 6% OV101. The response of each component was measured by area integration using a computerized data reduction system.

Results & Discussion

The nine compounds and their recoveries are listed in Table 1. The 1 l samples at high concentrations show good recoveries. The large variation of butyl carbitol acetate may be attributable to a data system error. Results for 3 l samples at 10 ug/l show large variations and a

Table I. Recoveries for selected organics from tap water for neutral pH extractions.

Name	1 l extraction - 2500 ug/l % Recovery[a]	3 l extraction 10 ug/l % Recovery[b]
methyl cellosolve acetate	79 ± 9	16 ± 0.3
styrene	99 ± 1	167 ± 25
anisole	119 ± 4	328 ± 20
phenol	48 ± 3	0
o-cresol	98 ± 4	105 ± 0.1
N,N-dimethyl aniline	108 ± 5	88[c]
benzothiazole	103 ± 4	27[c]
butyl carbitol acetate	86 ± 69	83[c]
2,6-dinitrotoluene	119 ± 53	217 ± 2

a = Values represent results of 3 replicate sample analyses

b = Values represent results of 2 replicate sample analyses.

c = No recovery in one sample, value is result where recovery was observed.

number of cases of no recoveries. The limiting factor for detection is most likely the use of packed column gas chromatography and could account for a large part of the variation. Recoveries at low levels, however, can be expected to be more variable due to the larger samples and extreme concentration factors required.

Conclusion

Extraction recoveries can be expected to be quite good at high component concentrations. At low levels, 10 ug/l, the variation will be larger and with packed column gas chromatography, may be unacceptable.*

*Note: GlassCapillary column gas chromatography (GC) was used for quantitation of survey samples lowering the effective GC detection limit by a factor of ca 10.

ATTACHMENT VIII

Sediments and Sludges Extraction Procedure---NEIC Aug. 1978

I. SCOPE & APPLICATION

1.1- Solids which precipitate or sediment out from various waters may be extracted for organic components analysis.

II. SUMMARY OF METHOD

2.1- An aliquot of sample is allowed to air dry. A portion of this is oven dried for percent dry weight calculation. The remainder is extracted in a Soxhlet extraction apparatus with 1:1 acetone:hexane, then concentrated/exchanged into iso-octane.

III. SAMPLE HANDLING & PRESERVATION

3.1- Samples are collected as grabs in wide mouthed glass containers with Teflon lined caps.

3.2- Samples are preserved by maintaining them at or below $4^{o}C$ during shipment and storage.

3.3- Extracts are stored in glass bottles with Teflon lined caps in explosion proof refrigerators at or below $4^{o}C$.

IV. DEFINITIONS & COMMENTS

4.1- While this procedure should be adhered to as closely as possible, sample characteristics (e.g., composition, amount available, and/or concentration of organics extracted) may require some deviation.

V. INTERFERENCES

5.1- Solvents and apparatus are potential sources of contamination.

Therefore, at least one "Reagent Blank" must be prepared and processed through the same analytical scheme as the associated samples.

5.2- Typical interferences from reagents include 4-methyl-4-hydroxy-2-pentanone (diacetone alcohol) from acetone, and phthalate esters from sodium sulphate.

VI. APPARATUS

6.1- Weighing: Mettler P1210N or equivalent, capable of taring 100 grams and weighing to $\pm$ 0.01 grams.

6.2- Glazed porcelain evaporating dishes for drying.

6.3- Laboratory oven capable of 105^{o}C constant heat.

6.4- Soxhlet extraction apparatus with cellulose thimble.

6.5- Kuderna-Danish evaporative condensor fitted with a three-ball Snyder column.

6.6- Drying column for extract.

6.7- Washing: All glassware should be washed thoroughly with Alconox, then rinsed with hot water and acetone.

VII. REAGENTS

7.1- Pesticide analysis grade acetone, hexane and 2,2,4-trimethyl-pentane (iso-octane).

7.2- Anhydrous sodium sulphate, washed with acetone just prior to use.

VIII. PROCEDURE

8.1- Drying:

8.1.1- Place an aliquot of sample (approximately 50 grams dry-weight) in a glass or glazed porcelain dish and allow to air dry in a

laboratory fume hood. The material should be stirred frequently until a free flowing powdery solid is obtained.

8.1.2- Determining percentage dry weight: Weigh out a 5-10 gram (if available) aliquot of air dried sample into a tared evaporating dish. Bake over night in an oven at 105°C and reweigh.

$$\% \text{ dry weight} = \frac{\text{sample weight after oven drying}}{\text{sample weight before oven drying}} \times 100$$

8.2- Extraction:

8.2.1- Weigh out a 30 gram (if available) portion of air dried sample and pulverize. Add water to yield an estimated 15% moisture content and mix thoroughly (average sludges will contain approximately 5% moisture after air drying).

8.2.2- Place sample in a cellulose extraction thimble and plug top with glass wool.

8.2.3- Extract using a Soxhlet extraction apparatus of appropriate size. Extract with 1:1 acetone:hexane for a minimum of 25 siphoning cycles.

8.3- Column Drying: Dry the extract by passing through a 15 x 3 cm column of acetone rinsed sodium sulphate. Wash the column into the filtrate thereafter.

8.4- Concentration

8.4.1- Concentrate the resulting solution in a Kuderna-Danish evaporative concentrator. Exchange into 4 ml of iso-octane, bringing the final volume up to 5 ml iso-octane.

8.4.2- Extracts containing suspected high concentrations of organics may be concentrated to higher volumes to avoid precipitation. Severe precipitation may be alleviated by diluting resulting concentrate with acetone.

IX. REFERENCES

9.1- "Draft Analysis of Sediment and Sludges for Priority Pollutants - Organics Parameters", April 1978, EPA Region VII.

ATTACHMENT IX

METHODS: VOLATILE ORGANICS ANALYSES
Purge and Trap - Gas Chromatography-Mass Spectrometry

This method is basically drawn from "Sampling and Analysis Procedures for Screening of Industrial Effluents for Priority Pollutants", U.S.E.P.A. Environmental Monitoring and Support Laboratory, Cincinnati, Ohio, 45268, March, 1977, revised April, 1977, and "Volatile Organic Compounds by GC/MS", U.S.E.P.A., NEIC, Denver, Colorado, 80225, July, 1978.

Scope

The Volatile Organics Analyses (VOA) method is designed to determine "priority pollutants" associated with the Consent Decree that are amenable to the purge and trap method. It is a gas chromatographic-mass spectrometric (GC-MS) method intended for the qualitative and quantitative determinations of these compounds.

The purge and trap method is complementary to the liquid-liquid extraction method. There is an area of overlap between the two methods, and some compounds may be analyzed by either method. The efficiency of recovery depends on the vapor pressure and water solubility of each compound. The overlap region in general consists of compounds which boil between 130° and 150°C (1 atmosphere pressure), with a water solubility of approximately two percent. The method of choice for these overlap region compounds is selected according to overall method efficiency and dependability.

Special Apparatus

Tekmar Liquid Sample Concentrator, Model LSC-1; Tekmar Company, P.O. Box 37202, Cincinnati, Ohio, 45222.

Special sorbent trap for LSC: stainless steel tube 1/8-inch O.D. by 17-cm.; packing from inlet, 1 cm glass wool, 5 cm. type 15 silica gel, 8 cm Tenax, 60/80 mesh; 3 cm. glass wool.

GC Column: a 6-ft. by 1/8-inch OD column packed with 0.2% Carbowax 1500 on 60/80 mesh Carbopack C; manufactured by Supelco, Supelco Park, Bellefonte, Pennsylvania, 16823.

Standards

For liquid standards, a primary standard solution for each compound was prepared from 10 ul of the compound in 10 ml of methanol. Concentrations were calculated from the desnity of each compound, and a standard mix was prepared by diluting a calculated volume of each solution (ca 150 ul) together to a total volume of 10 ml in methanol. Due to instability, acrolein

and acrylonitrile were prepared in a separate standard mix.

For gaseous standards - only vinyl chloride in this procedure - a primary standard solution was prepared by bubbling the gas into a tared volumetric flask of suitable solvent (methanol in this instance). The mass increment was measured and the concentration calculated. As with the liquid standards, a calculated volume was then diluted for the standard mix.

For internal standards, 100 mg each of bromochloromethane and 1,4-dichlorobutane were made up to 20 ml in methanol. For each day of analysis, 20 ul of this solution was diluted to 1.0 ml in water, and 10 ul of this preparation was added to each 5 ml sample aliquot, to give 200 ug/l of each component.

Analysis Procedure

The helium purge gas flow on a liquid sample concentrator (LSC) was adjusted to 40 ml/min. and the LSC valve set to the purge position. The VOA sample was removed from cold storage and brought up to ambient temperature. The bottle was carefully opened and the sample water poured into a 5-ml syringe to overflowing. The syringe plunger was replaced and the sample volume adjusted to 5.0 ml, and the syringe valve was closed. A 10 ul aliquot of the internal standard (IS) mixture was introduced into the sample by opening the valve and injecting the IS into the syringe. An 8-inch needle was attached to the syringe valve, and the sample was injected into the purging chamber of the LSC. The timer of the LSC was set to purge the sample for 12 minutes, with the silica gel-Tenax trap at ambient temperature (20-25^{o} C).

At this time, the oven of the gas chromatograph was brought to near ambient temperature by opening the oven door with the heater off.

After the 12-minute purge time the sample from the trap was injected into the GC by turning the valve to the desorb position and starting a timer for the analysis cycle (time zero). The GC-MS data collection was started at one minute; at four minutes the desorb was ended by turning the valve back to the purge position, and simultaneously the GC oven was closed and the oven temperature was set at 60^{o}C. The temperature program conditions: isothermal at 60^{o} until 8 minutes; program at 8^{o}C/mm to 170^{o}; hold at 170^{o} to the end of the program at 29 minutes.

After the sample purge, and while data was being collected, the trap was baked out at 210^{o}C for ten minutes, then allowed to cool to ambient temperature. Also, the sample tube was removed from the assembly, washed in methanol and baked out, and replaced on the LSC by a clean tube.

Mass Spectrometer Parameters

The mass spectrometer used was a Finnigan 1015 S/L interfaced to a Systems Industries System 150 data system. The operational parameters include: electron energy, 70 ev; mass range, 20-27 and 33-260 amu; integration time/amu, 17 milliseconds; samples/amu, 1.

GC Column Preparation

The column was connected at the inlet, the helium flow was adjusted, and the column was baked out overnight. This column must be handled with care, due to the fragile character of the Carbopack.

MS Calibration

The mass spectrometer was calibrated daily with perfluorotributylamine (FC 43), according to the Finnigan instrument manual. A further calibration check was made with the first run each day of analysis of a blank with internal standards added. The mass spectrum of bromochloromethane must meet these specifications:

m/e	Relative Intensity
49	100
130	65-98
128	50-75
51	25-35

Quality Assurance

The analysis of blanks is most important in the purge and trap technique, since the purging device and the trap can be contaminated by residues from very concentrated samples and by vapors in the laboratory. Blanks are of low-organic water, prepared by passing distilled water through an activated carbon column. If positive interferences are observed, the blank is repeated; if interferences persist, appropriate measures are taken to eliminate them before analyses are made.

The precision of the method is determined by running blanks dosed with the internal standards, bromochloromethane and 1,4-dichlorobutane. These compounds represent early and late eluters over the range of the Consent Decree compounds and are not on the list.

Each sample is dosed with the internal standards and analyzed by the set procedure. The operator monitors the sensitivity of the system to the internal standards as compared with blank runs; if the deviation is too great, a sample run is repeated. If excess deviation of sensitivity persists, appropriate

steps are taken by the operator to stabilize the operation.

To determine the precision of the method, replicate aliquots of environmental samples are analyzed, with at least one set of replicate analyses made for each group of 20 samples or less analyzed. Over the course of a survey, replicate analyses are made on samples which represent the entire range of concentrations and interferences found in that survey.

To determine the recovery of the method, at least one environmental sample for each group of 20 samples or less is reanalyzed after the addition of a spike mixture. The spike concentration should approximately double the background concentration. If the background is negligible, the spike concentration sould be five to fifteen times the lower detection limit.

The qualitative and quantitative determinations of the volatile priority pollutants are based upon the characteristic masses and their relative and absolute intensities, from which an extracted ion current profile is obtained for each compound. Details of these determinations are presented in "Computer-Assisted Evaluation of Volatile Organics GC/MS Data", NEIC, July, 1978.

ATTACHMENT X

Computer Assisted Evaluation of Organic Priority Pollutant GS/MS Data

NEIC - September 1978

1.0 Introduction

1.1 This procedure is applicable to GC/MS data collected under constant analytical conditions for the organic priority pollutant defined in "Sampling and Analysis Procedures for Screening of Industrial Effluents for Priority Pollutants". (1)

2.0 Summary of Method

2.1 GC/MS data files are processed by location of an internal standard that is used for response and retention time reference. Components of interest are then located by reverse searching from library spectra. If a compound is located and the match is sufficient, it is quantitated and its spectrum optionally printed. The concentrations are then calculated from each component found using a relative response quantitation technique. Printed reports of both quantitative and qualitative results are available.

3.0 Definitions and Comments

3.1 Unlike the 3 ion and retention time compound identification technique described for priority pollutant analysis in reference 1, this procedure allows the user to audit each identification where the spectra are printed. Thus, each identification is unambiquous and marginal data may be eliminated.

4.0 Interferences

4.1 In some cases, a spectrum may match the library reference sufficiently to be passed. During quantitation, however, the ion of interest may be too weak to locate and no entry will be made in the quantitation list. In such a case, no entry at all (e.g. no "not found" entry) will appear in the quantitation report. The name and match results will, however, appear in the qualitative data report.

4.2 Occasionally, multiple peaks will be detected during quantitation due to background interferences and multiple entries will be made in the quantitation list. Generally, the entry having the same label as the correct spectrum is used for quantitation and the others are disregarded. In some instances, however, the correct selection is not obvious and manual evaluation of the quantitation results must be done.

5.0 Apparatus

5.1 Finnigan INCOS data system software, Revision 3.1 or later. To initially setup this procedure, the user must understand and be proficient in the use of MSDS. (2)

6.0 Procedure

6.1 Procedure Setup

6.1.1 Load the procedures listed in Appendix I into the system disc or create the procedures from the trace of PPEVAL in Appendix II.

6.2 Library Setup

6.2.1 Build user libraries for each analytical class of priority pollutants (VOAs, base-neutrals and phenols). Appendicies III, IV and V are library lists of example libraries. The first entry must always be the internal standard and each entry must include the quantitation parameters and relative retention times.

6.2.2 Execute PPEVAL, edit the quantitation list for accuracy and update the library parameters using commands in "QUAN".

6.2.3 Using the "LIBR" program, generate hard copies of library spectra for reference. Using the library list editor, "EDLL", generate summaries of the entries and quantitation parameters as in Appendicies III, IV and V.

6.3 Routine Use

6.3.1 Analyze samples, standards and quality control samples using the same instrument conditions used to set up the libraries.

6.3.2 Using the namelist editor, create a namelist containing the names of the data files to be processed.

6.3.3 Execute the procedure as follows:

PPEVAL library, namelist, yes (no)

Where: library is the appropriate user library name.

namelist is the list containing the files to be processed.

yes (no) selects print out of the spectra at a peak that was identified by the procedure.

6.3.4 Appendix VI is an example of PPEVAL output for a sample containing one internal standard and one component. The "yes" option was selected.

7.0 Quality Control

7.1 Each identification can be manually audited if the "yes" option was selected. Inaccurate qualitative results may then be checked and manually corrected.

7.2 Quantitation data accuracy is monitored by use of standard quality control techniques such as daily standardization, replicate analysis and spikes. (3) Daily calibration of the method can be accommodated by analyzing the standard data first, updating the relative response factors, obtaining hard copy of the new factors (library list editor) and then analyzing sample data.

8.0 Precision and Accuracy

8.1 The overall precision and accuracy is limited to the quality of the raw data being processed.

9.0 References

(1) "Sampling and Analysis Procedures for Screening of Industrial Effluents for Priority Pollutants", US EPA, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio, March 1977, Revised April 1977.

(2) "INCOS Data System - MSDS Operators Manual - Revision 3", Finnigan Instruments, March 1978.

(3) "Quality Assurance Program for the Analyses of Chemical Constituents in Environmental Samples", US EPA, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio, March 1978.

Appendicies

I. List of procedures, file names, and functions for PPEVAL

II. Trace of PPEVAL

III. VOAs library list

IV. Base neutrals library list

V. Phenols library list

VI. Example PPEVAL output

PROCEDURE OR METHOD	FUNCTION
PPEVAL	INITIALIZATION
PPEVA	DATA FILE PROCESSING LOOP
PPEVB	DATA FILE PROCESSING
PPEVC	LOCATING THE INTERNAL STANDARD
PPEVD	INTERNAL STANDARD ERROR HANDLER
PPEVE	COMPOUND LOCATER
PPEVF	NOT DETECTED ERROR HANDLER
PPEVG	IDENTIFICATION CHECK
PPEVH	SPECTRA PRINTING
PRINP1	IDENTIFICATION REPORT HEADER
PRINP2	INTERNAL STANDARD ERROR MESSAGE

APPENDIX IIA.

```
TRACE OF PROCEDURE PPEVAL
   * ERASE
   * ;[********* PRIORITY POLLUTANT EVALUATION PROCEDURE *********]
   * ;[THIS PROCEDURE MAY BE USED TO EVALUATE GC/MS DATA          ]
   * ;[FOR PRIORITY POLLUTANT (EPA SECTION 307(A)) COMPOUNDS      ]
   * ;[THE PROCEDURE UTILIZES INTERNAL STANDARDS AND RELATIVE     ]
   * ;[RESPONSE FACTORS FOR QUANTITATION.  THE MSDS OPTION        ]
   * ;[SEARCH IS USED TO LOCATE AND IDENTIFY PEAKS.  THE EPA      ]
   * ;[IDENTIFICATION CRITERIA, E.G., THREE IONS PER COMPOUND     ]
   * ;[,IS USED TO LOCATE THE COMPOUND OF INTEREST.  MORE IONS    ]
   * ;[HOWEVER MAY BE USED AS THE FIT OF THE SEARCH ROUTINE WILL  ]
   * ;[YIELD MORE SPECIFICITY FOR THE COMPOUND.  THE FULL         ]
   * ;[SPECTRUM IS OUTPUT IN ORDER TO PROVIDE CONFIRMATION OF     ]
   * ;[THE PRESENCE OF THE COMPOUNDS.                             ]
   * ;[***********************************************************]
   * ;[TO USE PPEVAL. BUILD A LIBRARY CONTAINING THE SPECTRA OF   ]
   * ;[THE COMPOUNDS OF INTEREST.  INCLUDE THE QUANTITATIVE DATA  ]
   * ;[THAT IS NECESSARY AS DESCRIBED IN THE MSDS MANUALS.        ]
   * ;[CREATE A NAMELIST WITH THE NAMES OF THE FILES TO BE        ]
   * ;[PROCESSED.  EXECUTE THE PROCEDURE AS FOLLOWS:              ]
   * ;[    PPEVAL LIBRARYNAME, NAMELIST ,YES(NO)                  ]
   * ;[WHERE YES(NO) SELECTS PRINTED SPECTRA OF ACCEPTABLE        ]
   * ;[MATCHES.                  E.G. PPEVAL VO,SAMPLE            ]
   * ;[  WRITTEN 10AUG78 O.J.LOGSDON II  EPA-NEIC 303-234-4661    ]
   * ;[  REVISED 05SEP78 O.J.LOGSDON II  EPA-NEIC 303-234-4661    ]
   * ;SETS PPSCAN;EDLL YES(-;$;W;E);EDLL NO(-;W;E)
   * ;SETN $2;SET4 $1;PPEVA;FEED;BEEP;BEEP;BEEP
   *
   ERASE
   SETS PPSCAN
   EDLL YES (-;$;W;E)
   EDLL NO (-;W;E)
   SETN $2
   SET4 $1
   PPEVA
      * ERASE
      * ;[PART OF PROCEDURE PPEVAL                                  ]
      * ;[GET THE NEXT NAMELIST ENTRY AND CONTINUE PROCESSING       ]
      * ;[AT PPEVB                                                  ]
      * ;GETN;PPEVB;LOOP
      *
      ERASE
      GETN
      PPEVB
         * ERASE
         * ;[PART OF PPEVAL.  THIS PROCEDURE SETS THE LIBRARY ENTRY          ]
         * ;[POINTER TO THE FIRST ENTRY, WHICH MUST ALWAYS BE THE INTERNAL   ]
         * ;[STANDARD.  PPEVC IS THEN CALLED AND THE INTERNAL FOUND          ]
         * ;[THE SPECTRUM NUMBER OF THE INTERNAL STANDARD IS                 ]
         * ;[STORED IN I10 FOR FUTURE REFERENCE.  THE LIBRARY POINTER        ]
         * ;[IS THEN RESET TO THE BEGINNING, THE QUANTITATION LIST SET TO    ]
         * ;[THE FILE NAME AND EMPTIED OUT.  PPEVE IS CALLED TO LOCATE EACH  ]
         * ;[COMPOUND (IF PRESENT).  QUAN IS THEN CALLED TO CALCULATE        ]
         * ;[THE RESULTS AND THE PROCEDURE RETURNS TO PPEVA TO GET THE       ]
         * ;[NEXT FILE TO PROCESS.                                           ]
         * ;FILE(K PRIN.99/N;E)
         * ;EDLL PPLIST(-;W;E)
         * ;SET1 *1;PARA(I;H;E);CHRO(I;H1,1050,350;E);SET4 *1;PPEVC;SET10 I14;SET4 *0
         * ;SETO $1;EDQL(-;W;E);EDSL(-;W;E);SETL $3;PPEVE;QUAN(I;H;E)
         * ;EDLL PPLIST(8!1;E)
         * ;PRIN(@P1)
         * ;FILE(C PRIN.99/N,M:;E)
         * ;FEED
         * ;BEEP
         *
         ERASE
         FILE  (K PRIN.99/N;E)
         EDLL PPLIST (-;W;E)
         SET1 *1
         PARA  (I;H;E)
```

APPENDIX IIB.

```
CHRO  (I;H1,1050,350;E)
SET4 #1
PPEVC
   * ERASE
   * ;[PART OF PPEVAL                                          ]
   * ;[ROUTINE TO FIND AN INTERNAL STANDARD IN A SAMPLE         ]
   * ;[USE A REVERSE SEARCH TO LOCATE THE INTERNAL STANDARD     ]
   * ;SET14 #0
   * ;SEAR/V(I;$;V2500000;N2,10,600;&;D-60,60;E)
   * ;PPEVD
   *
   ERASE
   SET14
   SEAR  (I;$;V2500000;N2,10,600;&;D-60,60;E)/V
   PPEVD
      * IF PPEVD ,!14
      * ;[PART OF PPEVAL                ]
      * ;[NO INTERNAL STANDARD FOUND     ]
      * ;PRIN(@P2)
      * ;RETU PPEVB
      *
      IF PPEVD,!14
      PRIN  (@P2)
      RETU PPEVB
SET10 !14
SET4
SETQ $1
EDQL  (-;W;E)
EDSL  (-;W;E)
SETL $3
PPEVE
   * ;[PART OF PPEVAL                                          ]
   * ;[THIS ROUTINE LOCATES COMPOUNDS IN THE                   ]
   * ;[SAMPLE FILE BY COMPARING THE SPECTRA IN THE LIBRARY     ]
   * ;[WITH THE SAMPLE.  RELATIVE RETENTION TIMES ARE USED     ]
   * ;[AND REFERENCED TO THE INTERNAL STANDARD FOUND EARLIER.  ]
   * ;[THE LIBRARY POINTER IS BUMPED AND TESTED TO             ]
   * ;[SEE IF THE LAST LIBRARY ENTRY HAS BEEN PROCESSED.       ]
   * ;[THEN THE CURRENT SCAN NUMBER IS SET TO THE INTERNAL     ]
   * ;[STANDARD LOCATION BY RECALLING THE CONTENTS OF !10.     ]
   * ;[STORE THE SCAN NUMBER OF                                ]
   * ;[THE BEST MATCH IN VARIABLE 14 AND ALLOW INTEGRATION     ]
   * ;[AT THAT SPECTRUM NUMBER ONLY                            ]
   * ;[IF THE COMPOUND IS NOT FOUND, PLACE A NOT FOUND         ]
   * ;[ENTRY INTO THE QUANTITATION LIST FOR LATER REFERENCE    ]
   * ;SET4 !4,,#1
   * ;IF !24#1,!4
   * ;SET14 #0
   * ;SET1 !10
   * ;EDLL PPLIST($;W;E)
   * ;SEAR/V(I;$;%;V2500000;N1,10,10;D-20,20;E)
   * ;PRIN/KX(!4,2;!14,6;!15,6;!16,7;C;E)
   * ;PPEVF
   * ;LOOP
   *
   SET4 !4,,#1
   IF #1!24,!4
   SET14
   SET1 !10
   EDLL PPLIST ($;W;E)
   SEAR  (I;$;%;V2500000;N1,10,10;D-20,20;E)/V
   PRIN  (!4,2;!14,6;!15,6;!16,7;C;E)/KX
   PPEVF
      * [PART OF PPEVAL]
      * ;[IF THE FIT IS LESS THAN OR EQUAL TO 750   ]
      * ;[WRITE A NOT DETECTED, NAMED ENTRY INTO THE]
      * ;[QUANTITATION LIST FOR FUTURE REFERENCE    ]
      * ;PPEVG
      * ;EDQL(-;N;#;A;E)
      *
```

APPENDIX IIC.

```
            PPEVG
               * [PART OF PPEVAL                                    ]
               * ;[ACCESS ANY SCANS IDENTIFIED IN DETECT             ]
               * ;[AND INTEGRATE THEIR AREAS.  RECORD THE            ]
               * ;[DATA IN THE QUANLIST ASSIGNED EARLIER.            ]
               * ;[ALSO CHECK AND PASS ONLY PEAKS WITH               ]
               * ;[A FIT OF 750 OR GREATER                           ]
               * ;IF PPEVG !16,PPEVG #700
               * ;SET1 !14
               * ;CHRO(I;R;$;*;N1,3;A)>5,3;G-4,4;D-5,5;E)
               * ;PPEVH
               * ;RETU PPEVF
               *
               IF PPEVG!16,PPEVG#700
               SET1 !14
               CHRO  (I;R;$;*;N1,3;A)>5,3;G-4,4;D-5,5;E)
               PPEVH
                  * IF !26 PPEVH,PPEVH
                  * ;SPEC(*;N;H;E)
                  *
                  IF PPEVH!26,PPEVH
                  SPEC  (*;N;H;E)
               RETU PPEVF
            EDQL  (-;N;*;A;E)
         LOOP
      QUAN  (I;H;E)
      EDLL PPLIST (B11;E)
      PRIN  (@P1)
      FILE  (C PRIN.99/N,M:;E)
      FEED
      BEEP
   LOOP
FEED
BEEP
BEEP
BEEP
```

APPENDIX IID.

```
PRINP2.ME = C20;T;                                    PRIORITY POLLUTANT EVALUATION;
C;T;                    NO INTERNAL STANDARD WAS FOUND IN SAMPLE ;S1;
C;T;                                   ;D;F
;E
```

```
PRINP1.ME = C2;T;IDENTIFICATION REPORT          FILE:
;S1;C2;T;NO  SCAN  PURITY  FIT
;C;E
```

```
NU[redacted]ME
T  FORMULA                          RET TIME   BASE       AREA    U.P.#1    U.P.#2
.RET.TIME/CAS#         MASS  AMT.    REF.PEAK   RESP.FILE RESP.FACTOR

   1:      1,4-DICHLOROBUTANE (INTERNAL STANDARD)
5  C4.H8.CL2                        3:30       55        0.      0.000     0.000
        1.000       55.000200.00    VI      1            C         1.000

   2:      BROMOCHLOROMETHANE (INTERNAL STANDARD)
3  C.H2.CL.BR                       0:44       130       0.      0.000     0.000
        0.000       49.000200.00    VO      1            VS        1.000

   3:  02  ACROLIEN
5  C3.H4.O                          0:00       56        0.      0.000     0.000
        0.000       56.000200.00    VO      1            VS        1.000

   4:  03  ACRYLONITRILE
3  C3.H3.N                          0:00       53        0.      0.000     0.000
        0.000       53.000200.00    VO      1            VS        1.000

   5:  04  BENZENE
3  C6.H6                            2:19       78        0.      0.000     0.000
        0.000       78.000200.00    VO      1            VS        1.000

   6:  06  CARBONTETRACHLORIDE
2  C.CL4                            1:45       117       0.      0.000     0.000
        0.000       117.000200.00   VO      1            VS        1.000

   7:  06  CHLOROBENZENE
2  C6.H5.CL                         3:50       112       0.      0.000     0.000
        0.000       112.000200.00   VO      1            VS        1.000

   8:  10  1,2-DICHLOROETHANE
8  C2.H4.CL2                        1:26       62        0.      0.000     0.000
        0.000       62.000200.00    VO      1            VS        1.000

   9:  11  1,1,1-TRICHLOROETHANE
2  C2.H3.CL3                        1:41       97        0.      0.000     0.000
        0.000       97.000200.00    VO      1            VS        1.000

  10:  13  1,1-DICHLOROETHANE
9  C2.H4.CL2                        0:52       63        0.      0.000     0.000
        0.000       96.000200.00    VO      1            VS        1.000

  11:  14  1,1,2-TRICHLOROETHANE
'2 C2.H3.CL3                        2:32       97        0.      0.000     0.000
        0.000       83.000200.00    VO      1            VS        1.000

  12:  15  1,1,2,2-TETRACHLOROETHANE
'6 C2.H2.CL4                        3:27       83        0.      0.000     0.000
        0.000       83.000200.00    VO      1            VS        1.000

  13:  23  CHLOROFORM
8  C.H.CL3                          1:20       83        0.      0.000     0.000
        0.000       83.000200.00    VO      1            VS        1.000

  14:  29  1,1-DICHLOROETHENE
6  C2.H2.CL2                        0:28       61        0.      0.000     0.000
        0.000       96.000200.00    VO      1            VS        1.000

  15:  30  1,2-TRANS-DICHLOROETHENE
5  C2.H2.CL2                        1:01       96        0.      0.000     0.000
        0.000       96.000200.00    VO      1            VS        1.000

  16:  32  1,2-DICHLOROPROPANE
2  C3.H6.CL2                        2:11       63        0.      0.000     0.000
        0.000       63.000200.00    VO      1            VS        1.000

  17:  33A 1,3-CIS-DICHLORO-1-PROPENE
```

```
 1[redacted].H4.C[redacted]                     [redacted]           0.0[redacted] 0.000[redacted]
        0.000       [redacted]200.00[redacted]VO

VO  18:  33B 1,2-TRANS-DICHLORO-1-PROPENE
 110  C3.H4.CL2                     2:34       75        0.      0.000     0.000
        0.000       75.000200.00    VO      1            VS        1.000

VO  19:  38  ETHYLBENZENE
 106  C8.H10                        0:00       91        0.      0.000     0.000
        0.000       91.000200.00    VO      1            VS        1.000

VO  20:  44  METHYLENECHLORIDE
  84  C.H2.CL2                      0:04       84        0.      0.000     0.000
        0.000       84.000200.00    VO      1            VS        1.000

VO  21:  47  BROMOFORM
 250  C.H.BR3                       3:02       173       0.      0.000     0.000
        0.000       173.000200.00   VO      1            VS        1.000

VO  22:  48  BROMODICHLOROMETHANE
 162  C.H.CL2.BR                    1:59       83        0.      0.000     0.000
        0.000       83.000200.00    VO      1            VS        1.000

VO  23:  49  TRICHLOROFLUOROMETHANE
 136  C.CL3.F                       0:19       101       0.      0.000     0.000
        0.000       101.000200.00   VO      1            VS        1.000

VO  24:  51  DIBROMOCHLOROMETHANE
 206  C.H.CL.BR2                    2:32       129       0.      0.000     0.000
        0.000       129.000200.00   VO      1            VS        1.000

VO  25:  85  TETRACHLOROETHENE
 164  C2.CL4                        3:22       166       0.      0.000     0.000
        0.000       129.000200.00   VO      1            VS        1.000

VO  26:  86  TOLUENE
  92  C7.H8                         3:29       91        0.      0.000     0.000
        0.000       91.000200.00    VO      1            VS        1.000

VO  27:  87  TRICHLOROETHENE
 130  C2.H.CL3                      2:20       130       0.      0.000     0.000
        0.000       95.000200.00    VO      1            VS        1.000
```

FORMULA / RET.TIME/CAS#	MASS	AMT.	RET TIME REF.	BASE PEAK	AREA RESP.	FILE	U.P.#1 RESP.	FACTOR	U.P.#2
1: D10-ANTHRACENE (INTERNAL STANDARD)									
			3:44	188	44864.		0.000		0.000
1.000	188.000	20.00	BN	1		:S	1.000		
2: 01 ACENAPHTHENE									
C12.H10			2:39	154	0.		0.000		0.000
0.710	154.000	20.00	BN	1		:S	0.586		
3: 05 BENZIDINE									
C12.H12.N2			5:08	184	0.		0.000		0.000
1.345	184.000	50.00	BN	1		:S	0.047		
4: 08 1,2,4-TRICHLOROBENZENE									
C6.H3.CL3			1:18	180	0.		0.000		0.000
0.349	74.000	20.00	BN	1		:S	0.182		
5: 09 HEXACHLOROBENZENE									
C6.CL6			3:20	284	0.		0.000		0.000
0.893	284.000	20.00	BN	1		:S	0.264		
6: 12 HEXACHLOROETHANE									
C2.CL6			0:43	201	0.		0.000		0.000
0.192	117.000	20.00	BN	1		:S	0.398		
7: 18 BIS(2-CHLOROETHYL)ETHER									
C4.H8.O.CL2			0:37	93	0.		0.000		0.000
0.165	93.000	50.00	BN	1		:S	0.205		
8: 20 2-CHLORONAPHTHALENE									
C10.H7.CL			2:12	162	0.		0.000		0.000
0.589	162.000	20.00	BN	1		:S	0.612		
9: 25 1,2-DICHLOROBENZENE									
C6.H4.CL2			0:41	146	0.		0.000		0.000
0.183	146.000	20.00	BN	1		:S	0.706		
10: 26 1,3-DICHLOROBENZENE									
C6.H4.CL2			0:31	146	0.		0.000		0.000
0.138	146.000	20.00	BN	1		:S	0.519		
11: 27 1,4-DICHLOROBENZENE									
C6.H4.CL2			0:34	146	0.		0.000		0.000
0.152	146.000	20.00	BN	1		:S	0.895		
12: 35 2,4-DINITROTOLUENE									
C7.H6.O4.N2			2:59	165	0.		0.000		0.000
0.803	165.000	50.00	BN	1		:S	0.191		
13: 36 2,6-DINITROTOLUENE									
C7.H6.O4.N2			2:46	165	0.		0.000		0.000
0.744	165.000	50.00	BN	1		:S	0.184		
14: 37 1,2-DIPHENYLHYDRAZINE (MEAS. AS AZOBENZENE)									
C12.H10.N2			3:06	77	0.		0.000		0.000
0.834	77.000	50.00	BN	1		:S	1.066		
15: 39 FLUORANTHENE									
C16.H10			4:34	202	0.		0.000		0.000
1.223	202.000	20.00	BN	1		:S	0.714		
16: 40 4-CHLOROPHENYL PHENYL ETHER									
C12.H9.O.CL			2:59	204	0.		0.000		0.000
0.799	204.000	20.00	BN	1		:S	0.280		
17: 41 4-BROMOPHENYL PHENYL ETHER									
248 C12.H9.O.BR			3:20	250	0.		0.000		0.000
0.893	248.000	20.00	BN	1		:S	0.153		
BN 18: 42 BIS(2-CHLOROISOPROPYL)ETHER									
170 C6.H12.O.CL2			0:43	45	0.		0.000		0.000
0.193	45.000	50.00	BN	1		:S	0.664		
BN 19: 43 BIS(2-CHLOROETHOXY)METHANE									
172 C5.H10.O2.CL2			1:20	93	0.		0.000		0.000
0.359	93.000	50.00	BN	1		:S	0.586		
BN 20: 53 HEXACHLOROCYCLOPENTADIENE									
270 C5.CL6			1:20	237	0.		0.000		0.000
0.000	237.000	20.00	BN	1		:S	1.000		
BN 21: 54 ISOPHORONE									
138 C9.H14.O			1:09	82	0.		0.000		0.000
0.309	82.000	50.00	BN	1		:S	0.984		
BN 22: 55 NAPHTHALENE									
128 C10.H8			1:25	128	0.		0.000		0.000
0.379	128.000	20.00	BN	1		:S	1.287		
BN 23: 56 NITROBENZENE									
123 C6.H5.O2.N			1:07	77	0.		0.000		0.000
0.308	77.000	50.00	BN	1		:S	0.457		
BN 24: 62 N-NITROSODIPHENYLAMINE (MEAS AS DIPHENYLAMINE)									
169 C12.H11.N			3:12	169	0.		0.000		0.000
0.857	169.000	20.00	BN	1		:S	0.145		
BN 25: 63 N-NITROSODIPROPYLAMINE									
130 C6.H14.O.N2			0:55	70	0.		0.000		0.000
0.247	130.000	50.00	BN	1		:S	0.058		
BN 26: 66 DI-(2-ETHYLHEXYL)PHTHALATE									
398 C24.H38.O4			5:44	149	0.		0.000		0.000
1.536	149.000	20.00	BN	1		:S	0.041		
BN 27: 67 BUTYLBENZYLPHTHALATE									
312 C19.H20.O4			5:27	149	0.		0.000		0.000
1.468	149.000	20.00	BN	1		:S	0.581		
BN 28: 68 DI-N-BUTYLPHTHALATE									
278 C16.H22.O4			4:06	149	0.		0.000		0.000
1.098	149.000	20.00	BN	1		:S	1.732		
BN 29: 69 DI-OCTYLPHTHALATE									
390 C24.H38.O4			6:58	149	0.		0.000		0.000
1.866	149.000	20.00	BN	1		:S	0.580		
BN 30: 70 DIETHYLPHTHALATE									
222 C12.H14.O4			3:04	149	0.		0.000		0.000
0.821	149.000	20.00	BN	1		:S	0.953		
BN 31: 71 DIMETHYLPHTHALATE									
194 C10.H10.O4			2:40	163	0.		0.000		0.000
0.714	163.000	20.00	BN	1		:S	0.817		
BN 32: 72 BENZO(A)ANTHRACENE									
228 C18.H12			6:14	228	0.		0.000		0.000
1.670	228.000	20.00	BN	1		:S	0.120		
BN 33: 76 CHRYSENE									
228 C18.H12			6:14	228	0.		0.000		0.000
1.670	228.000	20.00	BN	1		:S	0.120		
BN 34: 77 ACENAPHTHYLENE									
152 C12.H8			2:33	152	0.		0.000		0.000
0.683	154.000	20.00	BN	1		:S	0.003		

[redacted] ANTH[redacted]

C14.H10 3:44 178 0. 0.000 0.000
1.000 178.000 20.00 BM 1 :S 1.433

36: 80 FLUORENE
C13.H10 3:00 166 0. 0.000 0.000
0.804 166.000 20.00 BM 1 :S 0.573

37: 81 PHENANTHRENE
C14.H10 3:44 178 0. 0.000 0.000
1.000 178.000 20.00 BM 1 :S 1.433

38: 84 PYRENE
C16.H10 4:34 202 0. 0.000 0.000
1.223 202.000 20.00 BM 1 :S 0.714

[redacted] FORMULA [redacted] R[redacted] A[redacted] U.P. [redacted] U.P.♦
REL.RET.TIME/CAS♦ MASS AMT. REF.PEAK RESP.FILE RESP.FACTOR

PH 1: D10-ANTHRACENE (INTERNAL STANDARD)
188 2:43 188 44864. 0.000 0.000
1.000 188.000 50.00 PH 1 :S 1.000

PH 2: 21 2,4,6-TRICHLOROPHENOL
196 C6.H3.O.CL3 1:46 196 0. 0.000 0.000
0.000 196.000100.00 PH 1 :S 0.461

PH 3: 22 4-CHLORO-3-METHYLPHENOL
142 C7.H7.O.CL 2:06 142 0. 0.000 0.000
0.000 142.000100.00 PH 1 :S 0.524

PH 4: 24 2-CHLOROPHENOL
128 C6.H5.O.CL 0:27 128 0. 0.000 0.000
0.000 128.000100.00 PH 1 :S 1.014

PH 5: 31 2,4-DICHLOROPHENOL
162 C6.H4.O.CL2 1:13 162 0. 0.000 0.000
0.000 162.000100.00 PH 1 :S 0.714

PH 6: 34 2,4-DIMETHYLPHENOL
122 C8.H10.O 1:11 122 0. 0.000 0.000
0.000 122.000100.00 PH 1 :S 0.617

PH 7: 57 2-NITROPHENOL
139 C6.H5.O3.N 0:37 139 0. 0.000 0.000
0.000 139.000100.00 PH 1 :S 0.534

PH 8: 58 4-NITROPHENOL
139 C6.H5.O3.N 5:01 139 0. 0.000 0.000
0.000 65.000100.00 PH 1 :S 0.000

PH 9: 59 2,4-DINITROPHENOL
184 C6.H4.O5.N2 2:53 184 543744. 0.000 0.000
1.000 184.000*1000. PH 1 :S 0.219

PH 10: 60 4,6-DINITRO-O-CRESOL
198 C7.H6.O5.N2 2:57 198 781312. 0.000 0.000
1.000 198.000*1000. PH 1 :S 0.319

PH 11: 64 PENTACHLOROPHENOL
264 C6.H.O.CL5 3:12 266 0. 0.000 0.000
0.000 266.000100.00 PH 1 :S 0.242

PH 12: 65A PHENOL
94 C6.H6.O 0:52 94 0. 0.000 0.000
0.000 94.000100.00 PH 1 :S 1.025

APPENDIX VIA.

QUANTITATION REPORT FILE: SMA5A

DATA: SMA5A.MI
0:00:00
SAMPLE: VOA STD MIX A W/I.S. SEPT 3, 1978
CONDS.:
FORMULA: INSTRUMENT: SYSIND WEIGHT: 0.000
SUBMITTED BY: ANALYST: ACCT. NO.:

AMOUNT=AREA * REF.AMNT/(REF.AREA* RESP.FACT)

NO	NAME
1	1,4-DICHLOROBUTANE (INTERNAL STANDARD)
2	BROMOCHLOROMETHANE (INTERNAL STANDARD)
3	02 ACROLIEN
4	03 ACRYLONITRILE
5	04 BENZENE
6	06 CARBONTETRACHLORIDE
7	07 CHLOROBENZENE
8	10 1,2-DICHLOROETHANE
9	11 1,1,1-TRICHLOROETHANE
10	14 1,1,2-TRICHLOROETHANE
11	15 1,1,2,2-TETRACHLOROETHANE
12	19 2-CHLOROETHYLVINYLETHER
13	23 CHLOROFORM
14	30 1,2-TRANS-DICHLOROETHENE
15	32 1,2-DICHLOROPROPANE
16	38 ETHYLBENZENE
17	44 METHYLENE CHLORIDE
18	47 BROMOFORM
19	48 BROMODICHLOROMETHANE
20	51 DIBROMOCHLOROMETHANE
21	85 TETRACHLOROETHENE
22	86 TOLUENE
23	87 TRICHLOROETHENE
24	88 VINYL CHLORIDE
25	29 1,1-DICHLOROETHENE

NO	M/E	SCAN	TIME	REF	RRT	METH	AREA	AMOUNT	%TOT
1	55	251	4:11	1	1.000	A BB	1191060.	200.000 PPB	4.55
2	49	75	1:15	1	0.299	A BB	1120890.	200.000 UG/L	4.55
3	NOT FOUND								
4	NOT FOUND								
5	78	175	2:55	1	0.697	A BB	1734110.	200.000 UG/L	4.55
6	117	139	2:19	1	0.554	A BB	1242110.	200.000 UG/L	4.55
7	112	272	4:32	1	1.084	A BB	1944750.	200.000 UG/L	4.55
8	62	117	1:57	1	0.466	A BB	1115510.	200.000 UG/L	4.55
9	97	134	2:14	1	0.534	A BB	1254820.	200.000 UG/L	4.55
10	83	189	3:09	1	0.753	A BB	806289.	200.000 UG/L	4.55
11	83	247	4:07	1	0.984	A BB	1293270.	200.000 UG/L	4.55
12	106	200	3:20	1	0.797	A BB	119982.	200.000 UG/L	4.55
13	83	103	1:48	1	0.430	A BB	1612750.	200.000 UG/L	4.55
14	96	88	1:28	1	0.351	A BB	774512.	200.000 UG/L	4.55
15	63	167	2:47	1	0.665	A BB	1008560.	200.000 UG/L	4.55
16	91	306	5:06	1	1.219	A BB	2419710.	200.000 UG/L	4.55
17	84	45	0:45	1	0.179	A BB	560656.	200.000 UG/L	4.55
18	173	221	3:41	1	0.880	A BB	1054980.	200.000 UG/L	4.55
19	83	153	2:33	1	0.610	A BB	1613140.	200.000 UG/L	4.55
20	129	189	3:09	1	0.753	A BB	1452530.	200.000 UG/L	4.55

NO	M/E	SCAN	TIME	REF	RRT	METH	AREA	AMOUNT	%TOT
21	129	243	4:03	1	0.968	A BB	1099630.	200.000 UG/L	4.55
22	91	251	4:11	1	1.000	A BB	1879520.	200.000 UG/L	4.55
23	95	178	2:58	1	0.709	A BB	999815.	200.000 UG/L	4.55
24	NOT FOUND								
25	96	63	1:03	1	0.251	M XX	55884.	200.000 UG/L	4.55

QUANTITATION FOR THIS COMPOUND MANUALLY ADDED ON

APPENDIX VIB.

NAM	NUM:	WT	FORMULA
VI	1:	126	C4.H8.CL2
VI	2:	128	C.H2.CL.BR
VI	3:	56	C3.H4.O
VI	4:	53	C3.H3.N
VI	5:	78	C6.H6
VI	6:	152	C.CL4
VI	7:	112	C6.H5.CL
VI	8:	98	C2.H4.CL2
VI	9:	132	C2.H3.CL3
VI	10:	132	C2.H3.CL3
VI	11:	166	C2.H2.CL4
VI	12:	106	C4.H7.O.CL
VI	13:	118	C.H.CL3
VI	14:	96	C2.H2.CL2
VI	15:	96	C2.H2.CL2
VI	16:	112	C3.H6.CL2
VI	17:	106	C8.H10
VI	18:	84	C.H2.CL2
VI	19:	250	C.H.BR3
VI	20:	162	C.H.CL2.BR
VI	21:	206	C.H.CL.BR2
VI	22:	164	C2.CL4
VI	23:	92	C7.H8
VI	24:	130	C2.H.CL3
VI	25:	62	C2.H3.CL

	NAME
	1,4-DICHLOROBUTANE (INTERNAL STANDAR
	BROMOCHLOROMETHANE (INTERNAL STANDAR
02	ACROLIEN
03	ACRYLONITRILE
04	BENZENE
06	CARBONTETRACHLORIDE
06	CHLOROBENZENE
10	1,2-DICHLOROETHANE
11	1,1,1-TRICHLOROETHANE
14	1,1,2-TRICHLOROETHANE
15	1,1,2,2-TETRACHLOROETHANE
19	2-CHLOROETHYLVINYLETHER
23	CHLOROFORM
29	1,1-DICHLOROETHENE
30	1,2-TRANS-DICHLOROETHENE
32	1,2-DICHLOROPROPANE
38	ETHYLBENZENE
44	METHYLENECHLORIDE
47	BROMOFORM
48	BROMODICHLOROMETHANE
51	DIBROMOCHLOROMETHANE
85	TETRACHLOROETHENE
86	TOLUENE
87	TRICHLOROETHENE
88	VINYL CHLORIDE

IDENTIFICATION REPORT FILE: D:SMASA.MI

NO	SCAN	PURITY	FIT
1	251	420	864
2	75	819	978
3	53	41	43
4	45	43	204
5	176	615	940
6	139	841	977
7	272	770	960
8	117	673	994
9	134	765	981
10	189	406	979
11	247	606	964
12	200	643	959
13	108	825	984
14	63 DCE	789	988
15	89	786	977
16	167	726	977
17	307	758	995
18	45	781	976
19	221	798	940
20	153	837	995
21	183	417	945
22	243	835	961
23	251	566	955
24	177	525	981
25	0	0	0

Spectra printouts deleted to conserve paper.

ATTACHMENT XI

Organic Compound Identification by Glass Capillary Gas Chromatography/Mass Spectrometry

1. Scope and Application

   1.1 This method is applicable to surface waters and industrial effluents.

   1.2 The limit of detection for this method varies from 1 to 10 ug/l (ppb) depending on the type of compound.

   1.3 The concentration range is from 1 to 100 ug/l (ppb).

2. Summary of Method

   2.1 Concentrated extracts of 1 to 3 liter water samples are injected into a glass capillary column gas chromatograph directly coupled to a quadrupole mass spectrometer thru a small diameter heated stainless steel glass lined tubing. A splitless injection technique is used. Initial identification is established using a routine computer search of a library of standard reference spectra. The identification is confirmed by comparing the mass spectra of reference standards, analyzed using the same instrumental conditions. The coincidence of the gas chromatography retention times of standards and sample components provides additional confirmation of identity.

3. Interferences

   3.1 Concentrated solvent extracts often contribute interferences and a method blank is always run to differentiate reagent contamination from sample components.

   3.2 Common solvent interferences are: diacetone alcohol (4-methyl-4-hydroxy-2-pentanone) from acetone, phthalates from sodium sulfate, and cyclohexene from dichloromethane.

4. Apparatus

   4.1 Finnigan Model 9500 gas chromatograph equipped with a glass capillary column.

      4.1.1 Grob type injector for splitless injection.

      4.1.2 Capillary glass column, 25 meters x 0.25 mm ID, OV-101.

4.2 Finnigan Model 3200 electron impact mass spectrometer.

4.2.1 Glass lined stainless steel tubing direct coupling to gas chromatograph.

4.3 Finnigan INCOS data system (1).

5. Procedure

5.1 Gas Chromatography

5.1.1 Inject 1 ul of sample into the gas chromatograph with the splitter turned off for 1 minute after injection then turn on. (Splitter flow 100 ml/min).

5.1.2 The initial column temperature is equilibrated at 60°C and held for 1 minute after injection, then a temperature program is initiated at 4°C/min. to a final temperature of 220°C and held from 10 to 15 minutes. Column flow is adjusted to give a nominal flow of 1.5 ml/min. at 100°C.

5.2 Mass Spectrometry

5.2.1 The following MS instrumental parameters are used:

Electron multiplier voltage	-	1600 volts
Lens voltage	-	100 volts
Collector voltage	-	35 volts
Extractor voltage	-	6 volts
Ion Energy voltage	-	10 volts
Electron Energy voltage	-	70 volts
Emission Current	-	0.5 ma

5.2.2 The following data acquisition parameters are used:

Scan time	- 2 sec.
Mass Range	- 33-300
Sensitivity	- 10-7 amp.

5.2.3 The data acquisition is initiated immediately upon injection of a sample into the gas chromatograph in a suspended mode with the ionizer turned off. At 4 minutes the ionizer is turned on and at 5 min. the data acquisition is changed from the suspended mode to the centroid mode and actual data collection begun. A normal analysis using the 25 meter capillary OV-101 column will require data collection for 35 to 40 minutes.

5.2.4 A reconstructed ion chromatogram is generated using the MSDS program system and specific spectra are then plotted. A manual computer search of the reference library gives an identification. The initial identification is then confirmed by comparison of sample spectra and reference spectra obtained by analyzing standards under the same instrumental conditions.

6. Quality Control

6.1 Daily calibration of the GC/MS is performed before any sample analysis using a standard reference compound. (Pufluorotributylamine-FC-43).

6.2 The reference compound is metered into the mass spectrometer via a variable leak valve at a constant rate. Several scans are recorded at a scan rate of 3 seconds and a sensitivity of 10-6 amps. The calibration is then made utilizing the MSDS system calibration routine.

6.3 An ion intensity ratio of 2 to 1 for mass 69 to mass 219 is desirable for good spectra using the capillary system. The ion intensity ratio can vary from 3 to 1 to almost 1 to 1 and still provide legitimate spectra.

7. References

(1) "INCOS Data System - MSDS Operators Manual - Revision 3", Finnigan Instruments, March 1978.

ATTACHMENT XII

COMPUTER ASSISTED EVALUATION OF ORGANICS CHARACTERIZATION GC/MS DATA

August 1978

1.0 This procedure is applicable to GC/MS data collected under constant analytical conditions for qualitative data analysis.

2.0 Summary of Method

2.1 GC/MS data files are processed by comparing spectra from the sample against spectra of known or suspected pollutants contained in a project related library. If a spectrum matches the project library spectrum sufficiently, an entry is made in a table showing at what spectrum number the match occured and how good the match was. After completion of the search for each spectrum in the project library, a list of the compounds searched for and the matching results is printed as well as each spectrum that was identified as a probable pollutant. If selected by the user, the procedure will then search the current version of the NB (EPA/NIH/MSDC) library attempting to identify unknown spectra from peaks selected by the Biemann-Billler algorithin in MAP.

3.0 Definitions and Comments

3.1 In some cases, compounds may be identified by comparison to external reference spectra only (1,2,3). These "unconfirmed" compound data may however be useful since the computer matching still traces the presence of selected compounds through each sample analyzed. Therefore, even these "unconfirmed" pollutants can serve to trace a waste stream.

3.2 Quantitation of pollutants identified is effected by locating the corresponding GC peaks on GC/FID (flame ionization detector) chromatograms. The areas or peak heights are measured and compared to the response of known amounts of pure standard compounds. The concentrations are then calculated. Since this scheme utilizes two chromatographic systems (GC/MS and GC/FID), in some cases, differences in these systems will allow identification by GC/MS but not allow quantitation. In such cases, "MS" is reported to signify a mass spectrometer identification.

3.3 The identities of some components are confirmed by the matching of their mass spectra and GC retention times to the data obtained from the analysis of a pure standard compounds. Such identities are indicated by "CF."

3.4 Components not identified by mass spectrometry are reported as "ND" to denote not detected.

3.5 Analytical schemes may not allow measurement of some suspected pollutants in all samples and the result is reported as "NA" or not analyzed.

4.0 Interferences

4.1 Since absolute GC retention times are used for setting the search windows, the windows must be wide enough to account for slight variations in instrument conditions. This could cause identification errors if compounds with similar spectra (isomers) are in the window. Manually checking each spectrum produced essentially eleminates any error.

) Apparatus

5.1 Finnigan INCOS data system software running revision 3.1 or later version. To initially set up this procedure, the user must understand and be proficient in the use of MSDS (4).

5.2 INCOS "NB" mass spectra library (5).

) Procedure

6.1 Procedure Setup

6.1.1 Load the procedures listed in appendix 1 onto the system disc or create the procedures from the trace of OCEVAL in Appendix 2.

6.2 Library Setup

6.2.1 Obtain spectra of the compounds of interest by running standards under the same analytical conditions to be used for sample analysis.

6.2.2 Using the library editor, create a library containing the standard spectra with chemical names and retention times. Obtain a reference spectrum of each library entry for a permanent record and reference via the library program:

G1; HS; G2; HS;... etc.

6.3 Routine use

6.3.1 Collect mass spectra of samples to be processed under the same conditions as the standards were analyzed.

6.3.2 Using the namelist editor, create a namelist containing the names of the files to be processed.

6.3.3 Execute the procedure:

OCEVAL library, namelist, no (yes)

Where: library is the user library name, namelist is the file containing the names of the data files to be processed and no or yes select a continued search through the NB library.

If the user wants only to perform an NB search, the procedure is initiated as follows:

OCEVAL NB, namelist

6.3.4 Appendix 3 is an example of OCEVAL output consisting of the following:

(1) The acquisition parameter listing

(2) A chromatogram with peaks labeled by MAP

(3) A list of the compounds being searched for and a summary of the search results.

(4) A collection of the spectra of peaks identified by the procedure

(5) Library matching results for peaks found by MAP but not identified in the user library.

7.0 Quality Control

7.1 Each identification is manually verified by comparing the sample spectrum to the reference spectrum in the user library. Inaccurate computer results are re-evaluated and the correct data reported.

8.0 Precision and Accuracy

8.1 The auto processing routine's accuracy for correctly identifying compounds is limited by the quality of the original GC/MS data.

9.0 References

(1) "Eight Peak Index of Mass Spectra," Mass Spectrometry Data Center, Aldermaston, Reading, UK. Second Edition 1974.

(2) "Registry of Mass Spectral Data," Stenhagen, Abramsson and McLafferty, Wiley & Sons, New York, 1974.

(3) "Atlas of Mass Spectra Data," edited by: Stenhagen, Abrahamsson and McLafferty, Wiley & sons, New York, 1969.

(4) "INCOS Data System - MSDS Operators Manual - Revision 3," Finnigan Instruments, March 1978

(5) "NBS - NIH/EPA/MSDC Library - Revision 3," Finnigan Instruments, March 31, 1978

APPENDIX I.

PROCEDURES AND METHODS REQUIRED FOR OCEVAL

1. OCEVAL
2. OCEV0
3. OCEV1
4. OCEV2
5. OCEV2A
6. OCEV2B
7. OCEV3
8. OCEV5
9. OCEV6
10. OCEV7
11. PRINO1.ME
12. PRINO2.ME

APPENDIX II. a.

```
TRACE OF PROCEDURE OCEVAL
   * [***** OCEVAL ********************** JULY 29,1978 *****]
   * ;[OCEVAL PROVIDES THE OPERATOR WITH A MEANS OF  ]
   * ;[LOCATING COMPOUNDS THAT ARE SUSPECT BASED ON ]
   * ;[THEIR RETENTION TIMES AND SPECTRA.  THESE]
   * ;[COMPOUNDS ARE SAVED IN A USER LIBRARY FOR]
   * ;[ACCESS BY OCEVAL.  IF DESIRED, THE USER MAY]
   * ;[ALSO SELECT THAT ALL OTHER PEAKS LOCATED BY MEANS]
   * ;[OF BILLER-BIEMANN IN MAP BE SEARCHED AGAINST THE]
   * ;[NB LIBRARY.  THE USER LIBRARY MUST CONTAIN]
   * ;[SPECTRA AND RETENTION TIMES.  ALSO, ALL DATA FILES]
   * ;[PROCESSED MUST HAVE SCANS AVAILABLE FROM 25]
   * ;[BELOW THE EARLIEST ELUTING COMPONENT (OR START AT 0)]
   * ;[TO 25 ABOVE THE LATEST ELUTING COMPONENT.]
   * ;[TO USER THE PROCEDURE. CREATE A LIBRARY]
   * ;[WITH THE SPECTRA AND RETENTION TIMES.  CREATE A]
   * ;[NAMELIST CONTAINING THE FILE TO BE PROCESSED.]
   * ;[                                          ]
   * ;[THEN:  >OCEVAL XY,NAMELIST,NO(YES)  ]
   * ;[                                          ]
   * ;[WHERE: XY IS THE USER LIBRARY NAME OR NB ]
   * ;[       NAMELIST IS THE NAMELIST CONTAINING THE FILES]
   * ;[            TO BE PROCESSED.
   * ;[  NO SELECTS NO NB LIBRARY SEARCH OR YES SELECTS]
   * ;[           AN NB SEARCH]
   * ;[           IF THE USER SELECTED THE NB LIBRARY]
   * ;[           INITIALLY NO ENTRY IS REQUIRED]
   * ;[LAST REVISED 9/27/78     OJLOGSDONII  ]
   * ;SET4 !1
   * ;EDLL YES(-;$;W;E);EDLL NO(-;W;E)
   * ;SETN OCTEMP;EDNL(-;$1;$2;W;E)
   * ;SET11 #0
   * ;OCEV0
   * ;BEEP;BEEP;BEEP
   * ;ERASE
   * ;[PROCEDURE OCEVAL IS COMPLETE]
   *
   SET4 !1
   EDLL YES (-;$;W;E)
   EDLL NO (-;W;E)
   SETN OCTEMP
   EDNL  (-;$1;$2;W;E)
   SET11
   OCEV0
      * SETN OCTEMP;SETN #0;GETN;SET4 $1
      * ;GETN;SETN $1;SETN !11;SET11 !11#1;GETN
      * ;OCEV1
      * ;SETL OCTEMP
      * ;EDLL(-;W;E)
      * ;FILE(K PRIN.99/1;E)
      * ;OCEV2
      * ;SET12 #0
      * ;SETS OCEV2;SETS #0
      * ;EDSL(-!12;W;E)
      * ;OCEV3
      * ;OCEV5
      * ;BEEP
      * ;LOOP
      *
      SETN OCTEMP
      SETN
      GETN
      SET4 $1
      GETN
      SETN $1
      SETN !11
      SET11 #1!11
      GETN
      OCEV1
         * PARA(I;H;E)
```

APPENDIX II. b.

```
      * ;SETS OCEV2;EDSL(-;W;E)
      * ;SETS OCEV1;EDSL(-;W;E)
      * ;MAP(I;F1;U100;V250000;33,300;N>2,5,7;H1,2000,500;E)
      *
      PARA  (I;H;E)
      SETS OCEV2
      EDSL  (-;W;E)
      SETS OCEV1
      EDSL  (-;W;E)
      MAP   (I;F1;U100;V250000;33,300;N>2,5,7;H1,2000,500;E)
   SETL OCTEMP
   EDLL  (-;W;E)
   FILE  (K PRIN.99/N;E)
   OCEV2
      * IF OCEV2 #25000,OCEV2 !24
      * ;OCEV2A
      * ;PRIN (@01)
      * ;EDLL (B!1;E)
      * ;PRIN (@02)
      * ;FILE (C PRIN.99,M:/N;E)
      * ;FEED
      *
      IF OCEV2#25000,OCEV2!24
      OCEV2A
         * SET4 !4,,#1;SET!4 #0
         * ;IF #1!24 OCEV2A,!4 OCEV2A
         * ;OCEV2B
         * ;LOOP
         *
         SET4 !4,,#1
         SET14
         IF OCEV2A#1!24,OCEV2A!4
         OCEV2B
            * EDLL($;W;E)
            * ;SEAR/V(I;$;&;V250000;N1,200,750;D-25,25;E)
            * ;PRIN/KX(!4,6;!14,5;!15,8;!16,6;C;E)
            * ;SETS OCEV2;EDSL(!14;W;E)
            * ;SETS OCEV1;EDSL(-!14;W;E)
            *
            EDLL  ($;W;E)
            SEAR  (I;$;&;V250000;N1,200,750;D-25,25;E)/V
            PRIN  (!4,6;!14,5;!15,8;!16,6;C;E)/KX
            SETS OCEV2
            EDSL  (!14;W;E)
            SETS OCEV1
            EDSL  (-!14;W;E)
         LOOP
      PRIN  (@01)
      EDLL  (B!1;E)
      PRIN  (@02)
      FILE  (C PRIN.99,M:/N;E)
      FEED
   SET12
   SETS OCEV2
   SETS
   EDSL  (-!12;W;E)
   OCEV3
      * GETS
      * ;SPEC(I;';T;H30,350;E)
      * ;LOOP
      *
      GETS
      SPEC  (I;';T;H30,350;E)
      LOOP
   OCEV5
      * SETL 93
      * ;OCEV6
      * ;SET4 N8
      * ;SETS OCEV1;SETS #0
      * ;OCEV7
```

APPENDIX II. c.

```
        * ;FEED
        *
        SETL S3
        OCEV6
           * IF OCEV6 #25000,OCEV6 !24
           * ;IF OCEV5 !26,OCEV5
           * ;RETU OCEV6
           *
           IF OCEV6#25000,OCEV6!24
           IF OCEV5!26,OCEV5
           RETU OCEV6
        SET4 NB
        SETS OCEV1
        SETS
        OCEV7
           * GETS
           * ;LIBR(I;';F;X1,3;HS;E)
           * LOOP
           *
           GETS
           LIBR  (I;';F;X1,3;HS;E)
           LOOP
        FEED
     BEEP
     LOOP
BEEP
BEEP
BEEP
ERASE
```

APPENDIX II. d.

```
PRINO1.ME = C;D;T;          ORGANICS CHARACTERIZATION REPORT          FILE:
;S1;C2;T;
;D;C2;E

PRINO2.ME = C2;T;     NUM SPEC#  PURITY  FIT
;C;E
```

APPENDIX III.

0/00/00 0:00:00 ORGANICS CHARACTERIZATION REPORT FILE: 0:0114593N.TI

0/00/00 0:00:00

NAM	NUM:	WT	FORMULA	NAME
39	1:	126	C8.H14.0	2-ETHYL-2-HEXENAL (NC)
39	2:	144	C9.H20.0	2,6-DIMETHYL-4-HEPTANOL OR 5 NONANOL (
39	3:	146	C6.H4.CL2	DICHLOROBENZENE ISOMER (NC)
39	4:	130	C8.H18.0	2-ETHYL-1-HEXANOL (NC)
39	5:	138	C9.H14.0	ISOPHORONE (NC)
39	6:	162	C8.H18.03	BUTYL CARBITOL (NC)
39	7:	0		POLY GLYCOL ETHER (NC UNKNOWN)
39	8:	134	C9.H10.0	1-PHENYL-1-PROPANONE (NC)
39	9:	154	C12.H10	BIPHENYL (NC)
39	10:	170	C12.H10.0	PHENYL ETHER OR HYDROXY BIPHENYL (NC)
39	11:	222	C12.H14.04	DIETHYL PHTHALATE (NC)
39	12:	0		POLY GLYCOL ETHER (NC UNKNOWN)
39	13:	220	C15.H24.0	2,6-DI-TERT-BUTYL-P-CRESOL (NC)
39	14:	96	C4.H4.0.N2	4(1H)-PYRIMIDINONE (NC)
39	15:	0		UNKNOWN PEAK A
39	16:	0		UNKNOWN PEAK B
39	17:	0		UNKNOWN PEAK C
39	18:	0		UNKNOWN PEAK D
39	19:	0		UNKNOWN PEAK E (A NITRILE?)
39	20:	154	C9.H10.03	1,2-BENZENEDIOL,4-(2-HYDROXYETHYL)-
39	21:	154	C10.H18.0	2-METHYL-1-NONEN-3-ONE (NC)
39	22:	266	C12.H27.04.P	TRIBUTYLPHOSPHATE (NC)
39	23:	268	C19.H40	PRISTANE (NC)
39	24:	140	C9.H16.0	3,3,5-TRIMETHYL-CYCLOHEXANONE (NC)
39	25:	154	C9.H14.02	2,2,6-TRIMETHYL-1,4-CYCLOHEXANEDIONE (NC
39	26:	162	C8.H18.03	BIS-(2-ETHOXYETHYL)ETHER (NC)
39	27:	222	C10.H22.05	2,5,8,11,14-PENTAOXAPENTADECANE (NC)

NUM	SPEC#	PURITY	FIT
1	0	0	0
2	0	0	0
3	0	0	0
4	221	446	856
5	0	0	0
6	0	0	0
7	0	0	0
8	0	0	0
9	0	0	0
10	0	0	0
11	0	0	0
12	0	0	0
13	0	0	0
14	0	0	0
15	696	411	854
16	696	395	856
17	928	652	974
18	0	0	0
19	0	0	0
20	0	0	0
21	480	361	824
22	602	572	943
23	0	0	0
24	0	0	0
25	0	0	0
26	267	201	830
27	0	0	0

APPENDIX D

Bacteriological Methods

Bacteriological Methods

Bacteriological analyses of fecal coliform bacteria densities were performed according to standard procedures using the Most Probable Number technique*. Using asceptic techniques, all samples were collected in sterile bottles prepared by the accepted procedure. Replicate analyses wereperformed for quality control purposes; these data showed very good control and are available in the NEIC laboratory files.

* Rand, M. et al, 1975. Standard Methods for the Examination of Water and Wastewater. 14th Ed. APHA - AWWA - SPCF, 1193 pp.

APPENDIX E

Bioassay Methods

BIOASSAY METHODS

Toxicity tests consisted of 96-hour bioassays performed according to EPA standardized methods (EPA-600143-78-012). A continuous flow proportional diluter was used which provided a series of six effluent concentrations and a 100% dilution water control. Test chambers were of all glass construction and of 8 liter capacity. Flow rates were regulated to provide a minimum of nine volumetric exchanges of test solution for each chamber for each 24-hour period. All concentrations were done in duplicate with all test chambers containing ten fish.

The test fish used were young of the year fathead minnows (Pimephales Promelas Refinesque) obtained from the Newtown Toxicology Laboratory located at Cincinnati, Ohio. The fish, approximately 4 cm long, were acclimated for 96-hours prior to testing in Kanawha River water and given prophylic treatment (25 mg/l Oxytetracycline HCl) to prevent bacterial infection.

Dilution water was obtained from the Kanawha River at a point approximately 3 km (2 River miles) upstream of the mouth of the Elk River. The dilution water was stored in 1100 liter (300 gallon) epoxy-coated wooden reservoirs and was replenished every 24-hours.

Test water for the South Charleston Sewage Treatment Company bioassay was pumped continuously and directly from outfall 001 to the bioassay laboratory. All test chambers were monitored daily for pH, temperature and dissolved oxygen concentration (Table E-1). In addition the high, middle and low concentrations were analyzed for total alkalinity. Water temperature in the test chambers was maintained at 23.5°C $\pm$ 1°C by use

of a constant temperature recirculating water bath.

Mortalities in each test chamber were recorded at 24-hour intervals. The 96-hour LC_{50} value was calculated by computerized tape program based on the Spearman-Karber probit technique.

TABLE E-1
Physical-Chemical Characteristics
Union Carbide South Charleston Sewage Treatment Company Effluent
August, 1978

Parameter	Effluent Concentration (%)						
	Control	10	18	32	56	75	100
				24-hour			
DO mg/l	6.7	6.4	6.0	6.0	5.7	5.8	5.7
Temp. °C	23.3	23.2	23.2	23.2	23.3	23.2	23.5
pH	7.3	7.4	7.5	7.6	7.7	7.8	7.8
Total Alkalinity	34				206		331
				48-hour			
DO mg/l	6.5	6.1	6.0	6.0	5.5	5.5	5.3
Temp. °C	23.6	23.5	23.5	23.4	23.7	23.7	24.0
pH	7.2	7.3	7.4	7.5	7.6	7.7	7.7
Total Alkalinity	29				155		236
				72-hour			
DO mg/l	6.9	6.5	6.5	6.2	5.7	5.5	5.5
Temp °C	23.6	23.5	23.4	23.4	23.5	23.5	23.8
pH	7.2	7.5	7.6	7.6	7.7	7.8	7.8
Total Alkalinity	28				198		328
DO mg/l	7.6	7.3	7.0	6.7	6.0	5.9	5.8
Temp. °C	23.4	23.4	23.5	23.4	23.4	23.7	24.1
pH	7.2	7.4	7.4	7.5	7.6	7.7	7.7
Total Alkalinity	30				225		357

APPENDIX F

Mutagen Assay Methods

Mutagen Assay Methods

I. Sample Extraction

A 4:1 (80% benzene, 20% isopropanol) mixture of solvents was placed in a clean, 1 gallon amber solvent bottle and continuously stirred during the extraction procedure to assure adequate mixing.

For basic extractions, one-liter portions of sampleswere adjusted above pH 12 with NaOH. Each one liter aliquot was extracted three times (5 minutes each) with 35 ml of fresh solvent. The solvent fraction was then separated, mixed with anhydrous sodium sulfate to remove any emulsion and filtered into a one-liter round bottom flask. The aqueous fraction was retained for acidic extraction.

The combined solvent fractions (35 ml x 3 liters of sample extracted) were evaporated to dryness at 50°C in a rotoevaporator. The residue was resuspended into 15 ml sterile dimethylsulfoxide (DMSO), labeled and stored at 4°C until assayed by the Ames Procedure.

II. Bacterial Mutagenicity Assay

The Standard Ames Bacterial Assay was performed using the plate incorporation assay as described by Ames, et al.* Acidic and basic sample extracts were screened with standard Salmonella typhimurium tester strains TA 98, TA 100, TA 1535 and TA 1537. Samples were first tested individually; if the sample demonstrated an elevated reversion rate a dose-response relationship between concentration of sample extract and number of revertant colonies was determined for each responsive tester strain. Samples exhibiting a negative mutagenic response were subjected to metabolic activation by addition of S-9 mix (supernated from 9000 x g centrifugation rat liver homogenate). The Bacterial Assay was then repeated as discussed above.

* Ames, B.N., McCann, J., and Yamasaki, E., Methods for Detecting Carcinogens and Mutagens with the Salmonella/Mammalian-Microsome Mutagenicity Test. Mutation Research, 31, (1975) 347-364.

III. Quality Control

A three-liter volume of sterile distilled water was added to a 1-gallon amber glass bottle and treated as a sample. This served as a blank on the sample bottles, distilled water, extracting solvents, emulsion removal, and the concentration process. A DMSO blank was tested to ensure that the material did not interfere with test results.

The tester strains, TA 1535, TA 1537, TA 98 and TA 100, were exposed to diagnostic mutagens to confirm their natural reversion characteristics. The strains were tested for ampicillin resistance, crystal-violet sensitivity, ultra-violet light sensitivity, and histidine requirement. Spontaneous reversion rates were tested with each sample analyzed.

Rat liver homogenate was tested with 2-aminofluorene against strains TA 98 and TA 100 to confirm the metabolic activation process.

Sterility checks were performed on solvents, extracts, liver preparation, and all culture media.

APPENDIX G

TECHNICAL INFORMATION

DATA BASE DESCRIPTION

TECHNICAL INFORMATION
DATA BASE DESCRIPTION

RTECS contains toxicity data for approximately 21,000 substances, but does not presently include all chemicals for which toxic effects have been found. Chemical substances in RTECS have been selected primarily for the toxic effects produced by single doses, some lethal and some non-lethal. Substances whose principal toxic effect is from exposure over a long period of time are not presently included. Toxic information on each chemical substance is determined by examining and evaluating the published medical, biological, engineering, chemical and trade information and data for each substance selected.

The Toxline data base contains over 650,000 records taken from material published in primary journals. It is part of the MEDLINE file from the National Library of Medicine and is composed of ten subfiles:

(1) Chemical-Biological Activities, 1965-
(taken from Chemical Abstracts, Biochemistry Sections)

(2) Toxicity Bibliography 1968-
(a subset of Index Medicus)

(3) Abstracts on Health Effects of Environmental Pollutants, 1971- (published by the American Society of Hospital Pharmacists)

(4) International Pharmaceutical Abstracts 1970-
(published by the American Society of Hospital Pharmacists)

(5) Pesticides Abstracts 1967-
(compiled by EPA

(6) Environmental Mutagen Information Center 1969-
(Dept. of Energy, Oak Ridge National Lab)

(7) Environmental Teratology Information Center 1950-
(Dept. of Energy, Oak Ridge National Lab)

(8) Toxic Materials Information Center
(Dept. of Energy, Oak Ridge National Lab)

(9) Teratology file 1971-1974
(a collection of citations on teratology compiled by the National Library of Medicine)

(10) The Hayes File on Pesticides
(a collection of more than 10,000 citations on the health aspects of pesticides compiled by Dr. W. J. Hayes, Jr., EPA)